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**INVESTIGATION AND EVALUATION OF MOTOR INSULATION  
FOR MULTIPLE RESTART APPLICATION**

**Second Phase Report**

**by**

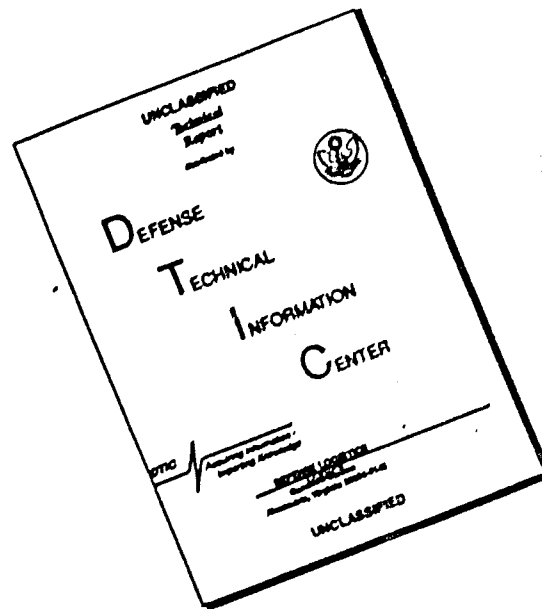
**W. Bradley  
Aerojet-General Corporation**

**TECHNICAL REPORT AFRPL-TR-67-104**

**April 1967**

**Rocket Propulsion Laboratory  
Research and Technology Division  
Air Force Systems Command  
Edwards, California**

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INVESTIGATION AND EVALUATION OF MOTOR INSULATION  
FOR MULTIPLE RESTART APPLICATION

SECOND PHASE REPORT

W. Bradley

PERIOD COVERED: 15 JAN 1967 THROUGH 1 APRIL 1967

FOREWORD

This second-phase technical report covers all work performed under Contract AF 04(611)-11609 from 15 January 1967 to 1 April 1967. The manuscript was released by the author on 7 April 1967 for publication as an RTD Technical Report.

The work on this contract by the Research and Technology Operations of the Aerojet-General Corporation, Sacramento, California, is being accomplished under the technical direction of Lt. R. Schoner (RPMC), Air Force Flight Test Center, Rocket Propulsion Laboratory, Edwards Air Force Base, California, 93523.

W. Bradley is the project manager for this program. Dr. T. N. Throckmorton conducted the statistical correlations. Work relating to the effect of ingredients on performance and properties was conducted by A. A. Stenersen.

Publication of this report does not constitute Air Force approval of the findings or conclusions. It is published only for the exchange and stimulation of ideas.

ABSTRACT

The primary purpose of this program is to investigate the properties and behavior of elastomeric insulation materials during multiple restart conditions and the influence of these properties on materials performance.

During the second phase of work, a correlation analysis was conducted to determine which virgin and charred material properties were significantly related to the performance of five representative materials during one, two, and three pulse motor firings. These properties were then determined on ten additional materials. All 15 materials were reviewed primarily on the basis of these properties, and four primary candidates (V62, V44, 9790V1-126K and USR 3800) were selected for further evaluation in pulse-motor firings (5, 12, and 21 pulses). A correlation analysis was also conducted to establish the relationship of the fillers, additives, and chemical ingredients to firing performance and significant properties.

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SECTION I

INTRODUCTION

This program was initiated under the sponsorship of the Air Force Rocket Propulsion Laboratory. The primary objective was to investigate the properties and behavior of elastomeric insulation during transient (heating and cooling) and steady-state heating conditions, and the influence of these properties on materials performance. A second objective was to develop a technique for predicting insulation performance for any stop-start duty cycle.

The program was divided into the following three phases of work:

- Phase I, Laboratory Investigations
- Phase II, Analytical Study
- Phase III, Verification Testing

The purpose of Phase I, Laboratory Investigations, was to (1) determine the properties of elastomeric insulation that exert the greatest influence on performance; (2) establish which available compositions exhibit the best performance; and (3) determine what insulation ingredients affect performance.

The purpose of Phase II, Analytical Study, was to develop an analytical technique for predicting insulation performance in a multistart environment.

The purpose of Phase III, Verification Testing, was to verify the performance predicted for the best materials determined in Phase I by test firing three motors.

The initial work on this program was described in the First Phase Report, AFRPL-TR-67-33, and covered a portion of the work to be accomplished in Phase I, Laboratory Investigations. The present report presents the tasks completed from 15 January 1967 to 1 April 1967. This completes the work planned in Phase I.

## SECTION II

### SUMMARY

This report presents the results of progress made during the period from 15 January 1967 to 1 April 1967, under Contract AF 04(611)-11609, and includes the following:

#### A. SELECTION OF SIGNIFICANT PROPERTIES

A correlation analysis was conducted relating the thermal, chemical, physical, and structural properties of five materials (V-44, V-62, 40SA40, SMR 81-8, and 970VI-126K) to the performance of these same materials during pulse motor firings. The analysis and accompanying technical appraisal of the results indicate that heat capacity, thermal diffusivity, thermal conductivity, and heat of combustion of the virgin material were significantly related to performance. Properties of the char were not found to be significant because of the loss of char that took place upon ignition of the next pulse. Results of the correlation analysis and motor firings indicate that, in addition to good thermal and ablative properties, consideration has to be given to other factors such as heat-soak stability of the virgin materials and susceptibility of char to separate from the virgin material during shut-down.

#### B. TESTING FOR SIGNIFICANT PROPERTIES

Heat capacity, density, thermal diffusivity, thermal conductivity, and heat of combustion properties were determined for the ten remaining materials in the program--N 356, MX-4737, USR 3800, V-61, V-50, V-51, SD 850-15C, 40SA2, SMR 81-15, and USR 3804. Data from these tests were then used as described below to select the best materials.

#### C. SELECTION OF BEST MATERIALS

Four prime candidates (9790VI-126K, V-62, V-44, and USR 3800) and three secondary candidates (USR 3804, SD 850-15C, and N 356) were selected for use at a later date in 5- and 12-pulse verification motor firings. This selection was accomplished by using the significant property data and plasma-arc data for the 15 candidate materials, together with other material characteristics such as processability, service life, storage stability, and demonstrated capability.

D. DETERMINATION OF INFLUENCE OF INGREDIENTS ON PERFORMANCE AND PROPERTIES

A correlation analysis was conducted establishing the relationship of the filler, additives, and chemical ingredients of five materials (V-44, V-62, 40SA40, BMR 81-8, and 9790VI-126K) to the performance and significant properties of these same materials. An analysis of the results indicate weight percent carbon, weight percent oxygen, C/O ratio and O/H ratio of the virgin material along with weight percent carbon and  $\text{SiO}_2$  in the char residue were related to performance. The data were insufficient to distinguish between the effects of different forms of carbon, oxygen, and hydrogen that might be present in the materials. A cross correlation of ingredients and properties showed a relationship between weight percent carbon, weight percent oxygen, weight percent total filler, C/O ratio, and O/H ratio of the virgin materials and the significant properties.

SECTION III

TECHNICAL DISCUSSION

The scope of the work discussed in this report includes (1) establishing which of the thermal, chemical, physical, and structural properties of five representative elastomeric insulation materials correlated with their performance during motor firings; (2) conducting tests for the properties that did correlate on ten additional candidate materials; (3) selecting materials considered to be the best for multiple restart solid motor applications; and (4) determining the influence of ingredients in the five elastomeric insulation materials on performance and properties.

A. SELECTION OF SIGNIFICANT PROPERTIES (TASK C, PHASE I)

In a previous task in Phase I (Task A), the fifteen candidate elastomeric insulation materials shown in Figure 1 were selected for evaluation in the program. Thermal, physical, and chemical properties along with motor firing performances were also obtained in Tasks A and B on five materials considered as being representative of their groups--V-44, V-62, 40SA40, SMR 81-8, and 9790VI-126K. The properties that were investigated are shown in Figure 2. It was the purpose of this task to determine which of the properties correlated with firing performance in order to establish the properties that were significantly related to performance.

This task was accomplished by conducting a single correlation analysis incorporating the 80 variables shown in Figure 3. Heat of combustion and density properties for the heat soaked materials (materials heat soaked for 5 and 30 minutes at 400°F) were not significantly different than those for the original virgin material and, accordingly, were not included in the correlation analysis. The correlation coefficients for those properties that had significant relationships with plasma arc and one or more of the motor performance tests are shown in Figure 4. The confidence interval was set at 95%, producing a critical correlation coefficient of 0.88. A positive correlation factor indicates an increase in ablation rate or regression rate as the property increases, and vice versa. Additional correlation coefficients down to the 90% confidence level are also given to indicate data which show a trend towards correlations. Because of the small sample size involved (five), there are certain limitations in using a simple correlation analysis for selecting significant properties. The use of this approach must be looked upon purely as an arbitrary screening device subject to an overall technical appraisal of the results. (See Appendix I for a further discussion.)

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<u>Material Code</u>	<u>Elastomer</u>	<u>Filler</u>	<u>Supplier</u>
<u>NBR Class</u>			
Gen-Gard V-44	Butadiene-Acrylonitrile	Asbestos-Silica	General Tire & Rubber Co.
N-356	NBR-Phenolic	Inorganic Hydrate	B. F. Goodrich
MX-4737	NBR-Phenolic	Silica	Fiberite Corp.
US 3800 (Mod)	Low Temperature NBR-Phenolic	Boric Acid	U. S. Rubber Co.
Gen-Gard V-50	Low-Temperature NBR	Silica-Asbestos	General Tire & Rubber Co.
Gen-Gard V-51	Low-Temperature NBR	Silica	General Tire & Rubber Co.
Gen-Gard V-61	Epoxy-Polysulfide NBR	Asbestos	General Tire & Rubber Co.
850-1SC	PBAN-Epoxy (Trowelable)	Asbestos Carbon Black Antimony-Trioxide	Aerojet-General Corp.
<u>SBR Class</u>			
Gen-Gard V-62	SBR-Phenolic	Silica	General Tire & Rubber Co.
<u>Urethane Class</u>			
40SA 2	Urethane	Silica Potassium-Titanate	American Polytherm Co.
40SA 40	Urethane	Silica Potassium-Titanate	American Polytherm Co.
<u>Butyl Class</u>			
SMR 81-8	Butyl	Asbestos-Silica	Stoner Rubber Co.
SMR 81-15	Butyl	Asbestos Potassium-Titanate Silica	Stoner Rubber Co.
<u>EPR Class</u>			
9790VI-126K	Ethylene-Propylene	Silica	General Tire & Rubber Co.
USR 3804	Ethylene-Propylene	Silica	U. S. Rubber Co.

Figure 1. Prospective Elastomeric Materials

<u>Virgin Material</u>	<u>Virgin Material Heat Soaked</u>	<u>Char Layers</u>
Kinetic Studies (TGA)	Kinetic Studies (TGA)	Density
Differential Thermal Analysis (DTA)	Differential Thermal Analysis (DTA)	Permeability
Density	Density	Pore Spectra
Thermal Conductivity	Thermal Conductivity	Thermal Conductivity
Heat Capacity	Heat Capacity	Heat Capacity
Heat of Combustion	Heat of Combustion	Thermal Expansion
Pyrolysis Gases	Pyrolysis Gases	Emissivity
Regression Rates	Weight Loss	Reflectivity
Tensile Strength and Elongation	Tensile Strength and Elongation	Shear Strength
		Compressive Strength
		Tensile Strength
	<u>Degradation Products</u>	
	Density	
	Heat Capacity	
	Thermal Conductivity	
	Viscosity	
	Heat of Cracking	
	Heat of Formation	

Figure 2. Properties to be Investigated

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## Virgin Material

Apparent Activation Energy, TGA  
 Apparent Specific Rate Constant at 1000°F, TGA  
 Seat of Formation  
 Density, 100°F, 250°F, 300°F  
 Thermal Conductivity, RT, 150°F, 250°F  
 Heat Capacity, 150°F, 250°F, 300°F  
 Heat of Combustion  
 Tensile Strength  
 Diffusivity, RT, 150°F, 250°F  
 Plasma Arc Ablation, 50, 100, 225 Btu/ft<sup>2</sup>-sec  
 Plasma Arc Regression, 50, 100, 225 Btu/ft<sup>2</sup>-sec

## Virgin Material, Heat Soaked 5 Min at 400°F

Thermal Conductivity, RT, 150°F, 250°F  
 Heat Capacity, 150°F, 250°F, 300°F  
 Tensile Strength  
 Weight Loss  
 Diffusivity, RT 150°F, 250°F

## Charred Material

Density  
 Pore Spectra  
 Shear Strength  
 Compressive Strength  
 Tensile Strength  
 Emissivity, 1325°F, 1555°F  
 Heat Capacity, 500°F, 932°F, 1652°F  
 Thermal Expansion, 212°F, 932°F, 1652°F  
 Thermal Conductivity, 200°F, 500°F, 700°F  
 Diffusivity, 200°F, 500°F, 700°F  
 Permeability

## Gases

Density  
 Heat of Formation

## Motor Performance

Ablation Motor 1 - 50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Ablation Motor 2 - 50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Ablation Motor 3 - 50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Regression Motor 1 - 50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Regression Motor 2 - 50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Regression Motor 3 - 50, 100, 225, 400 Btu/ft<sup>2</sup>-sec

Figure 3. Variables for Correlating Properties and Motor Performance



[illegible]

# Report AFRPL-TR-67-104

Motors Ablation				Correlation Motors Regression															
Motor 2				Motor 3				Motor 1				Motor 2				Motor 3			
0	225	400		50	100	225	400	50	100	225	400	50	100	225	400	50	100	225	400
	(0.96)	0.95																	
	(0.86)	0.95																	
				(-0.82)															
				(0.81)	(0.80)														
81)	0.88	(0.86)					(0.80)				(0.85)				0.98				
	0.96	0.99				0.90	0.86			(0.84)	(0.80)			0.88					
80)	(-0.83)			(-0.84)	-0.96					-0.96	(-0.84)			-0.96					
89	(-0.83)			(-0.82)	-0.93	-0.89				-0.90	-0.98	-0.89		-0.92					
82)				-0.89	(-0.83)					-0.89	-0.94	(-0.81)		-0.88					
91	0.96	0.90		(0.83)	(0.83)	0.89	(0.81)			0.88	0.95			(0.82)	0.93				
	0.95	0.96				(0.87)				(0.88)				0.89					
				(-0.82)												(-0.87)	-0.88	(-0.83)	
				-0.94														-0.98	
				(-0.83)								(-0.86)	(-0.84)			-0.96		-0.91	
														(-0.87)					(0.82)
								-0.96								(-0.84)	(-0.87)		
				0.90														0.97	
				0.91														(-0.85)	
	0.98					0.94													
91	(0.87)						0.99												

Figure 4. Correlation of Properties with Ablation and Regression Performance

2

III, A, Selection of Significant Properties (Task C, Phase I) (cont.)

On the basis of the above analysis, the following virgin material properties were selected for testing on the remaining ten materials in Task D (see paragraph B, below):

1. Density (not significant but needed for thermal conductivity calculations)
2. Heat capacity
3. Diffusivity (heat soaked 5 min at 400°F)
4. Thermal conductivity (heat soaked 5 min at 400°F—calculated from 1, 2, and 3 above)
5. Heat of combustion

It is to be noted that properties of the char such as diffusivity, heat capacity, density, and permeability were not found to be significant in the pulse motors. One explanation for this is the loss of char that took place upon ignition of the next pulse, which would reduce the overall effect of char properties on performance. However, as these properties should influence performance to some degree, they will be obtained on the three best materials along with thermogravimetric analysis data and pyrolysis data for use in Phase II when the performance of materials in the Verification Motors is predicted. The loss of char mentioned above resulted in higher ablation rates during two/and three-pulse motor firings than during the single-pulse firing (see First Phase Report). The current work on multipulse application has therefore indicated that, in addition to good thermal and ablation properties, consideration has to be given to factors such as heat-soak stability of virgin materials and susceptibility of char to separate from the virgin material during shutdown.

The correlation of the four significant virgin material properties with motor performance are graphically presented in Figures 5 to 8. These figures show (1) that as the heat capacity and heat of combustion of the virgin materials increased, the ablation rates decreased and (2) as the thermal diffusivity and thermal conductivity of the heat soaked materials increased, the ablation rates increased. The best overall correlation of data appears to have been obtained on the three-pulse motor at the 225 Btu/ft<sup>2</sup>-sec heat flux level.

A two variable equation describing the ablation rates for the three-pulse motor as a function of thermal conductivity and heat of combustion

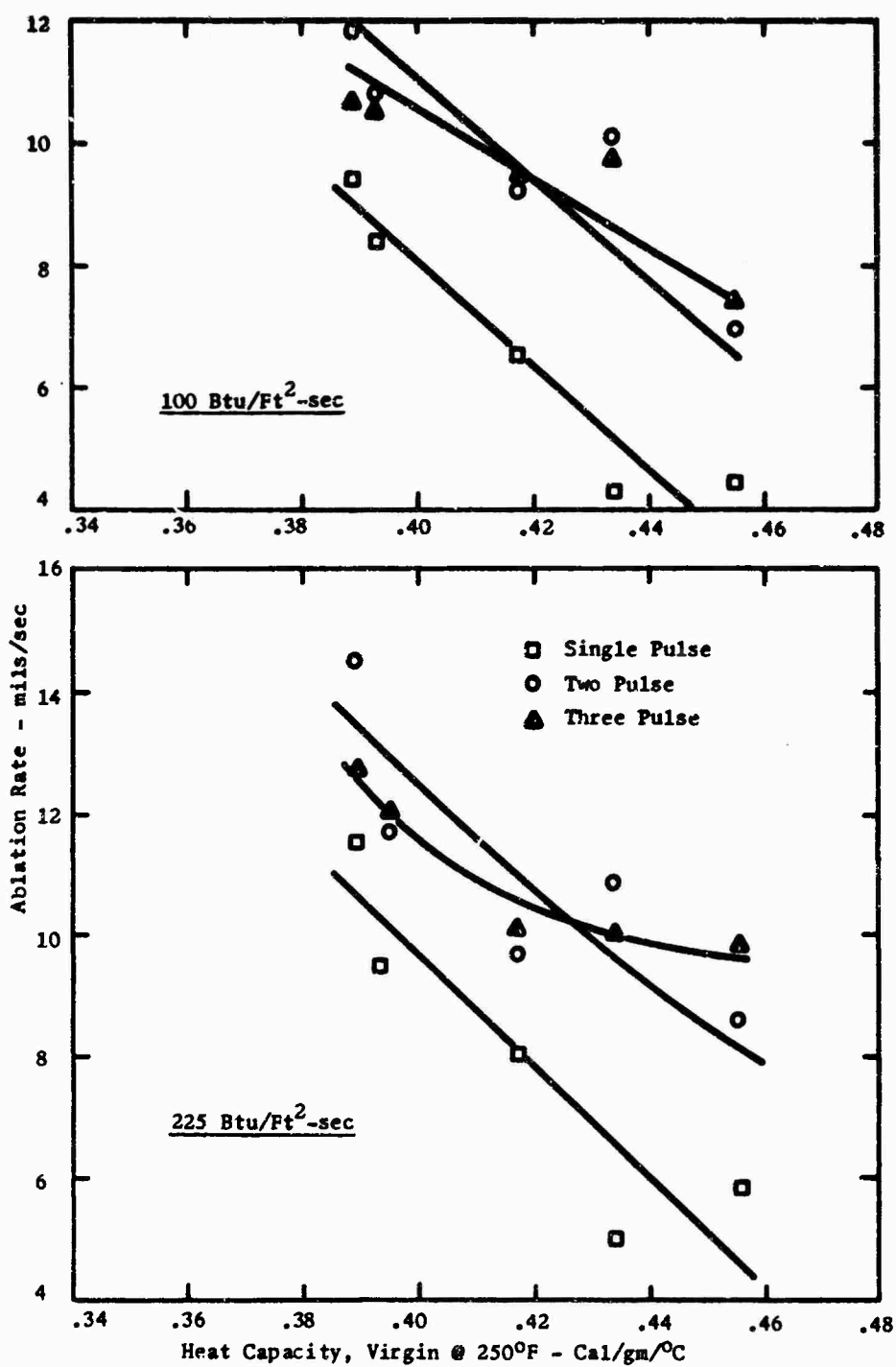


Figure 5. Ablation Rate vs Heat Capacity

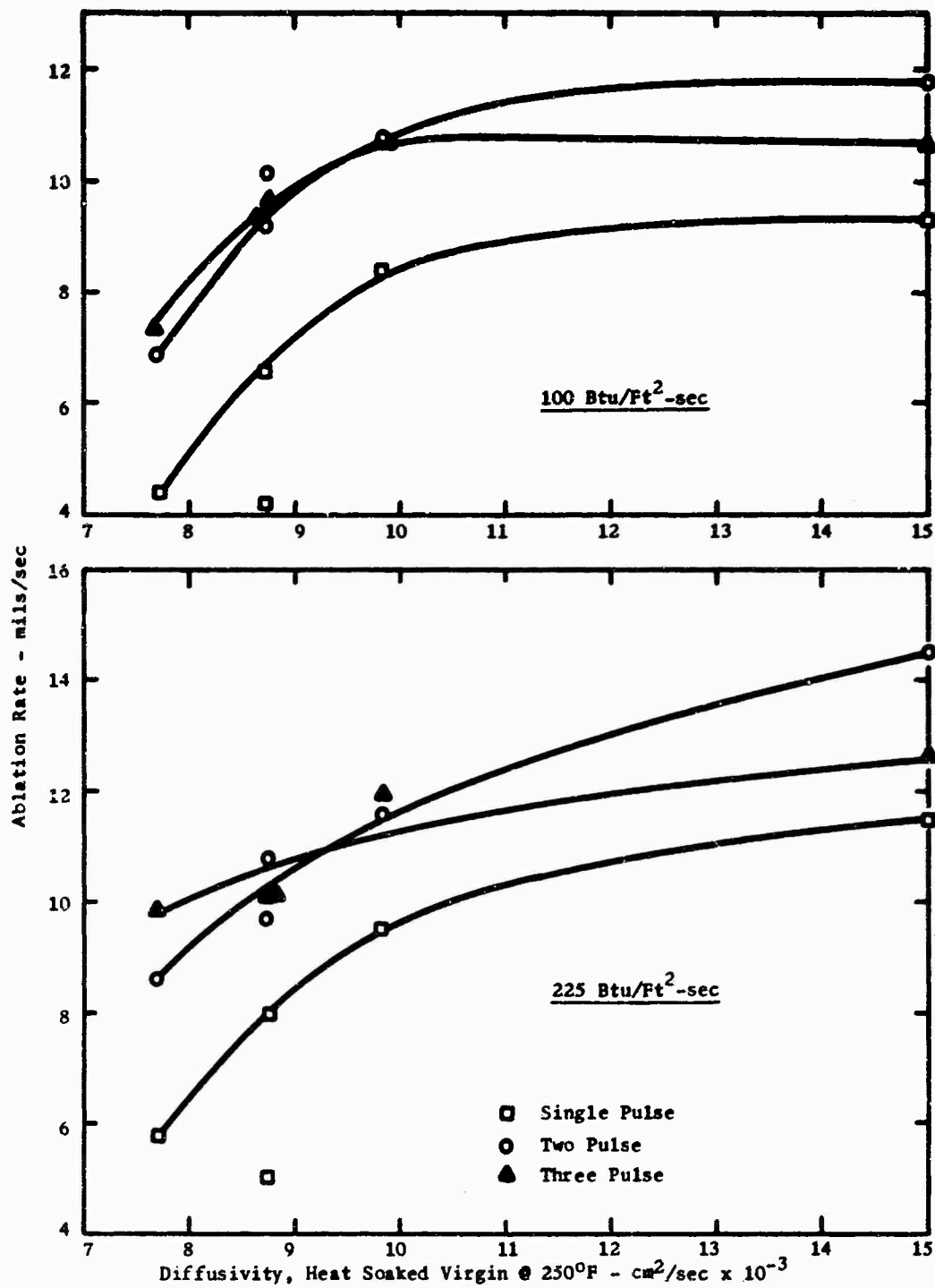


Figure 6. Ablation Rate vs Diffusivity

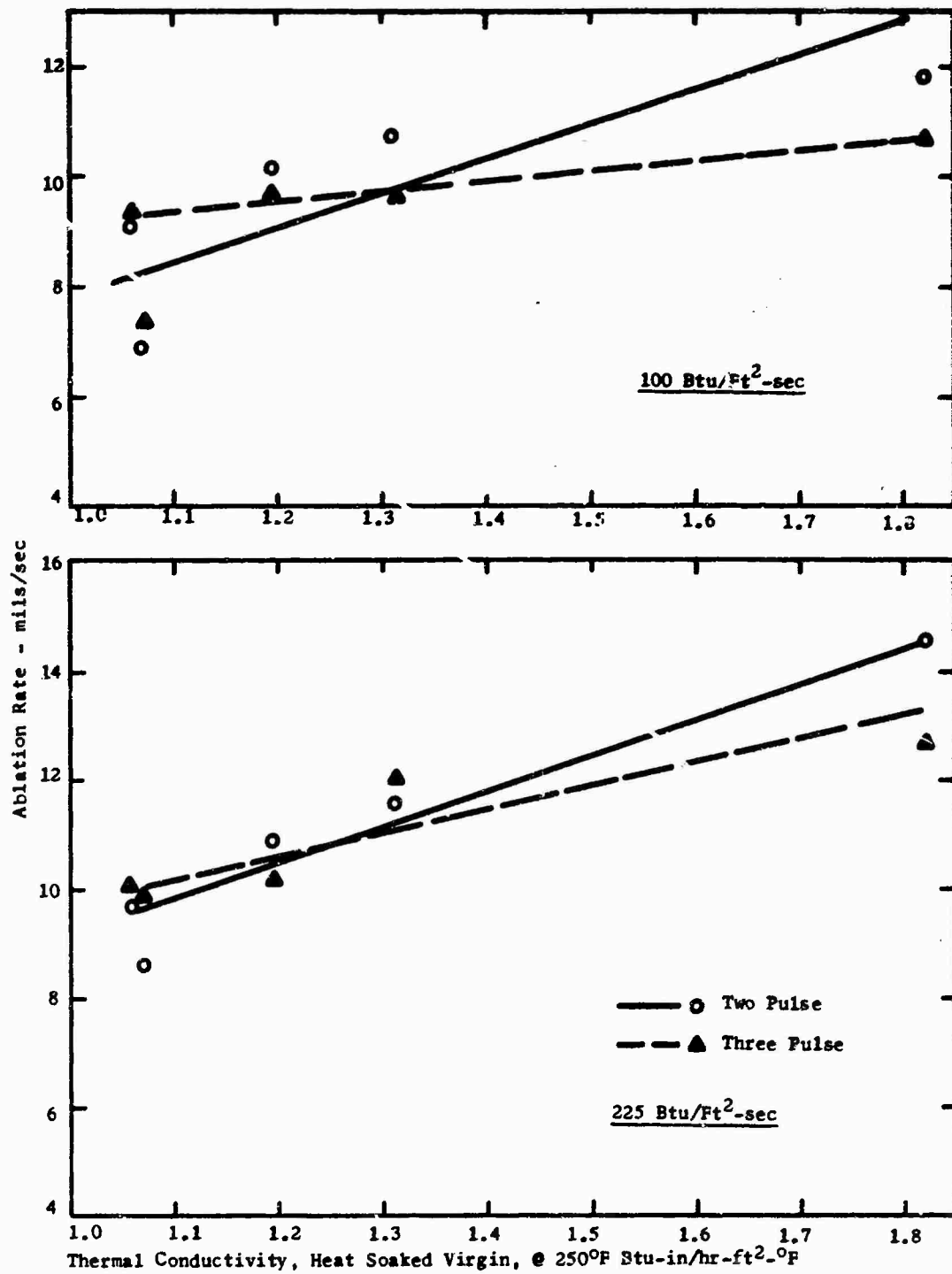


Figure 7. Ablation Rate vs Thermal Conductivity

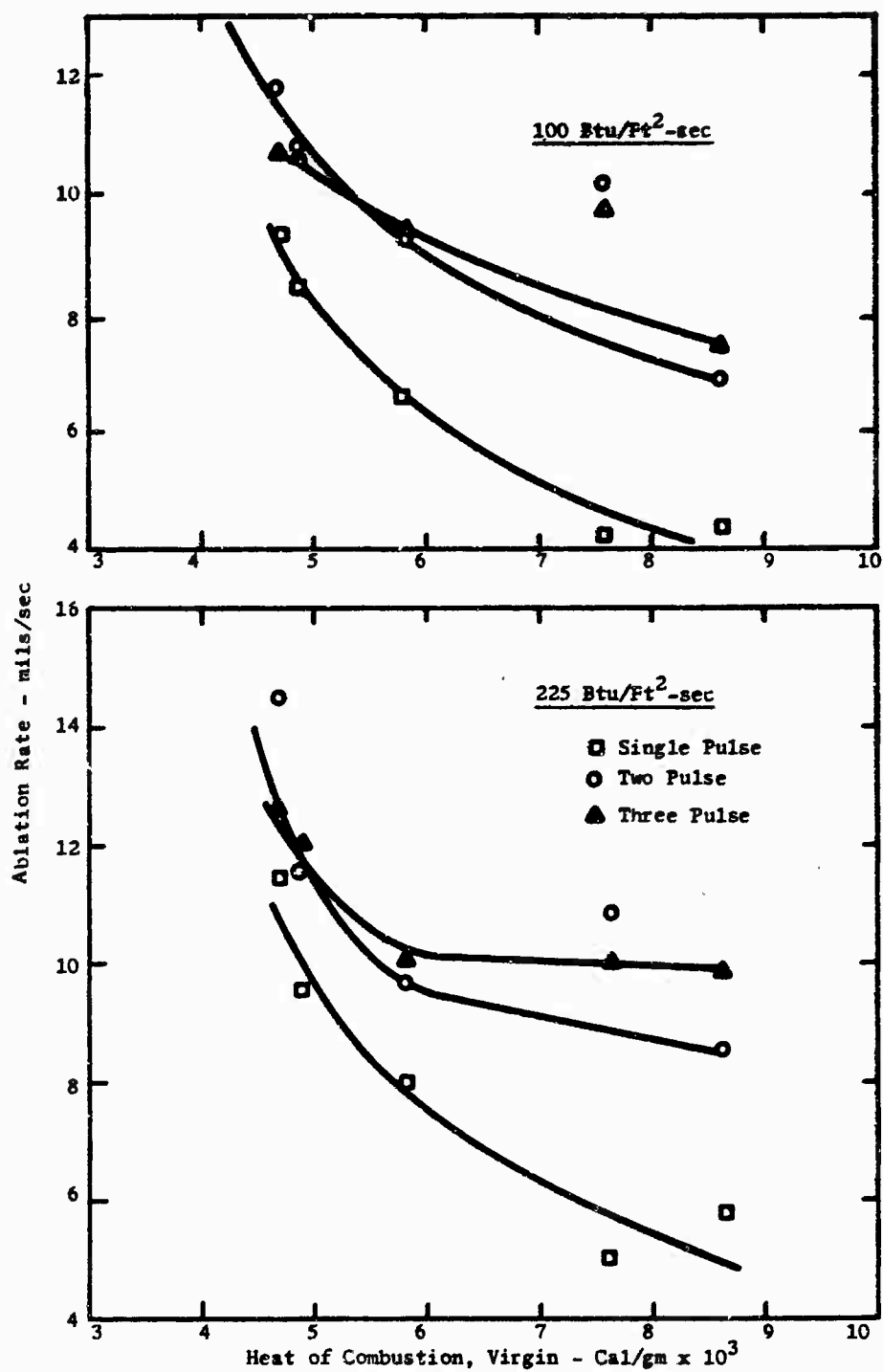


Figure 8. Ablation Rate vs Heat of Combustion

### III, A, Selection of Significant Properties (Task C, Phase I) (cont.)

was derived through use of the multiple regression analysis approach outlined in Appendix I. This equation is as follows:

$$\dot{a} = 9.650 + 2.593K - 0.0003216C$$

where

$\dot{a}$  = ablation rate, mils/sec of Motor 3 at 225 Btu/ft<sup>2</sup>-sec

K = thermal conductivity at 250°F, Btu-in./hr-ft<sup>2</sup>-°F

C = heat of combustion, cal/gm

The above equation, together with plasma-arc test data was used in Task F (see paragraph C, below) to select the best materials for use in the Verification Motors (Phase III). The significance of the plasma-arc tests is apparent from Figures 4 and 9, which show that the ablation data obtained during plasma-arc testing correlated well with motor ablation performance, particularly at the 225 Btu/ft<sup>2</sup>-sec heat-flux level. An equation describing the ablation rates for the three-pulse motor as a function of plasma arc rates, was also derived as follows:

$$\dot{a} = 5.79 + 1.30X$$

where

$\dot{a}$  = ablation rate, mils/sec of Motor 3 at 225 Btu/ft<sup>2</sup>-sec

X = plasma-arc ablation rate at 225 Btu/ft<sup>2</sup>-sec

Equations for regression were not derived, since it appears from Figure 4 that there was insufficient correlation between properties and regression and little correlation between motor ablation and regression. This fact would indicate that, at least for the five materials involved, surface regression has less effect on overall performance during pulse-motor operation than is normally attributed to it for single-pulse operation, undoubtedly because of the previously mentioned loss of char.

### B. TESTING FOR SIGNIFICANT PROPERTIES (TASK D, PHASE I)

In this task, the ten remaining materials, N356, MX 4737, USR 3800, V50, V51, V61, SD 850-15C, 40SA2, SMR 81-15 and USR 3804, were tested for density, heat capacity, heat of combustion, thermal diffusivity, and thermal



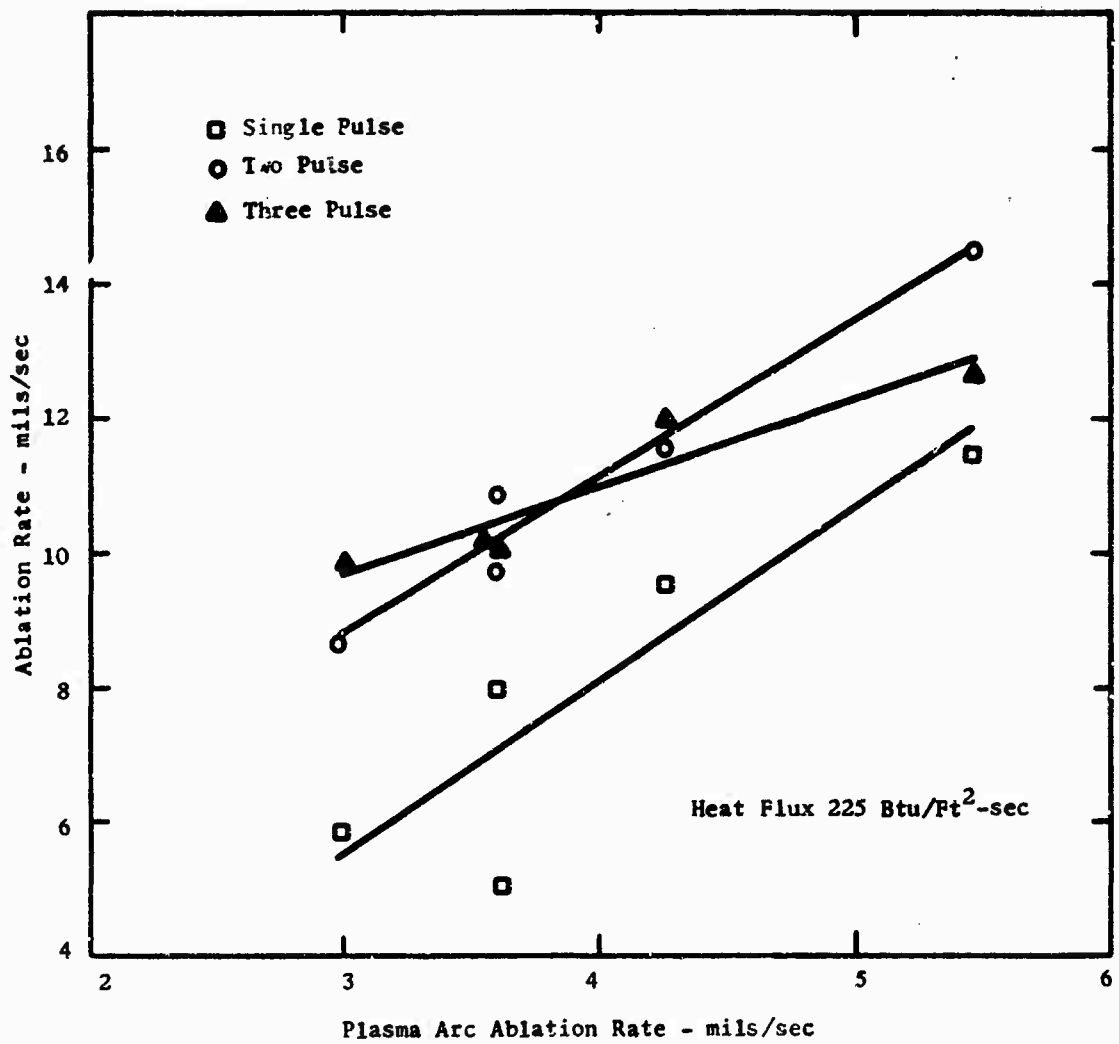


Figure 9. Motor Ablation Rate vs Plasma Arc Ablation Rate

III, B, Testing for Significant Properties (Task D, Phase I) (cont.)

conductivity. The test procedures used were identical to those previously outlined in Report AFRPL-TR-67-33, which covered the first phase of work. Results of the testing are discussed below:

1. Density

The density data for the virgin materials are tabulated in Figure 10 and graphically presented in Figure 11. The densities of the original five materials are also given in Figure 11 for comparison. These data show that MX 4737 and SD 850-15C are the heaviest materials and V62 and 9790V1-126K are the lightest.

2. Heat Capacity

The heat capacity data for the virgin materials are given in Figure 10 and graphically presented in Figure 12 along with the heat capacities of the original five materials. These data show that 9790V1-126K, V62, and USR 3800 have the highest heat capacity.

3. Heat of Combustion

The heat of combustion data for the virgin materials are given in Figure 10. Comparable data for the original five materials are V44-5822, V62-8633, 40SA40-4762, SMR 81-8-4866, and 9790V1-126K-7549 cal/gm. These data show that V62, 9790V1-126K, and USR 3804 have the highest heats of combustion.

4. Thermal Diffusivity

Results of the thermal diffusivity tests are shown in Figure 10. Complete data were obtained on materials that had been heat soaked for 5 minutes at 400°F. Partial data were also obtained on virgin materials to show the extent of change resulting from heat soak. In all cases, the heat-soaked materials had lower diffusivities than the virgin materials. Results of the heat-soak tests are presented in Figure 13, along with data on the original five materials.

The large change observed in the majority of the diffusivity curves leads to some speculation that experimental error had entered into the testing. Because the diffusivity calculation is a function of the thickness squared, any expansion or contraction of the material during testing could contribute significantly to erroneous results. This possibility was not accounted for while testing. However, as the majority of the materials exhibited the same relative change in diffusivity, the results should not affect the overall selection of the best materials.

<u>Property</u>	<u>N 356</u>	<u>MK 4737</u>	<u>USR 3800</u>	<u>V50</u>	<u>V51</u>
<u>Density (GM/CC)</u>					
T, °F	100°-1.206 <u>1.203</u>	101°-1.393 <u>1.385</u>	101°-1.146 <u>1.146</u>	100°-1.249 <u>1.247</u>	101°-1.1 <u>1.1</u>
Ave	1.204	1.389	1.146	1.248	1.1
T <sub>2</sub> , °F	210°-1.153 <u>1.143</u>	209°-1.358 <u>1.358</u>	206°-1.092 <u>1.087</u>	202°-1.213 <u>1.214</u>	217°-1.1 <u>1.1</u>
Ave	1.148	1.358	1.090	1.214	1.1
T <sub>3</sub> , °F	248°-1.124 <u>1.099</u>	301°-1.335 <u>1.332</u>	250°-0.999 <u>0.942</u>	300°-1.177 <u>1.177</u>	302°-1.0 <u>1.0</u>
Ave	1.112	1.333	0.971	1.177	1.0
<u>Heat Capacity (Cal/GM/°C)</u>					
150°F	0.3920 <u>0.4148</u>	0.3258 <u>0.3169</u>	0.4075 <u>0.4095</u>	0.3750 <u>0.3510</u>	0.4 <u>0.3</u>
Ave	0.4034	0.3214	0.4085	0.3630	0.3
200°F	0.4632 <u>0.4466</u>	0.3661 <u>0.3716</u>	0.4841 <u>0.4650</u>	0.4026 <u>0.4176</u>	0.3 <u>0.3</u>
Ave	0.4549	0.3689	0.4746	0.4101	0.3
250°F	0.4600 <u>-</u>	0.3538 <u>0.3635</u>	0.4495 <u>0.4724</u>	0.4082 <u>0.3937</u>	0.4 <u>0.4</u>
Ave	0.4600	0.3586	0.4610	0.4010	0.4
300°F	Melted	0.3458 <u>0.3459</u>	Melted	0.3844 <u>0.3826</u>	0.4 <u>0.4</u>
Ave		0.3459		0.3835	0.4
<u>Heat of Combustion (Cal/GM)</u>					
	6348	4483	6474	5812	6

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	<u>V51</u>	<u>V61</u>	<u>SD 850-15C</u>	<u>408A2</u>	<u>SMR 81-15</u>	<u>USR 3804</u>
49	101°-1.164	102°-1.306	101°-1.387	101°-1.324	101°-1.368	101°-1.198
47	<u>1.165</u>	<u>1.309</u>	<u>1.392</u>	<u>1.329</u>	<u>1.368</u>	<u>1.200</u>
48	1.165	1.308	1.390	1.327	1.368	1.199
13	217°-1.122	209°-1.244	213°-1.347	209°-1.298	222°-1.331	198°-1.166
14	<u>1.124</u>	<u>1.223</u>	<u>1.354</u>	<u>1.296</u>	<u>1.331</u>	<u>1.166</u>
14	1.123	1.233	1.351	1.297	1.331	1.166
77	302°-1.090	296°-1.151	298°-1.318	296°-1.252	305°-1.304	302°-1.126
77	<u>1.091</u>	<u>1.142</u>	<u>1.319</u>	<u>1.255</u>	<u>1.305</u>	<u>1.125</u>
77	1.091	1.146	1.319	1.254	1.305	1.126
750	0.4065	0.3210	0.3366	0.3446	0.3329	0.4062
510	<u>0.3896</u>	<u>0.3501</u>	<u>0.3507</u>	<u>0.3500</u>	<u>0.2932</u>	<u>0.3676</u>
630	0.3981	0.3355	0.3437	0.3473	0.3131	0.3869
026	0.3918	0.4115	0.3336	0.3654	0.3588	0.4056
176	<u>0.3917</u>	<u>0.3980</u>	<u>0.3430</u>	<u>0.3628</u>	<u>0.3603</u>	<u>0.4100</u>
101	0.3918	0.4048	0.3383	0.3641	0.3596	0.4078
082	0.4090	0.4350	0.3589	0.3794	0.3516	0.4050
937	<u>0.4033</u>	<u>0.4264</u>	<u>0.3626</u>	<u>0.3707</u>	<u>0.3616</u>	<u>0.4052</u>
010	0.4062	0.4307	0.3608	0.3750	0.3566	0.4051
844	0.4154	0.4008	0.3439	0.3790	0.3369	0.4063
826	<u>0.4018</u>	<u>0.3942</u>	<u>0.3408</u>	<u>0.3781</u>	<u>0.3427</u>	<u>0.3997</u>
835	0.4086	0.3975	0.3423	0.3786	0.3398	0.4030
812	6592	5441	6151	4784	4699	7053

Figure 10. Significant Properties of Ten Remaining Materials

2

<u>Property</u>	<u>N 356</u>	<u>MK 4737</u>	<u>USR 3800</u>	<u>V50</u>	
<u>Diffusivity, Virgin Material - Heat Soaked 5 Min at 400°F (CM<sup>2</sup>/sec)</u>					
RT	0.00110	0.00104	0.00093	0.00113	0.
125°F	0.00057	0.00109	0.00077	0.00123	0.
200°F	0.00067	0.000802	0.00079	0.00104	0.
275°F	0.00063**	0.00109**	0.00062**	0.00087	0.
<u>Diffusivity, Virgin Material (CM<sup>2</sup>/sec)</u>					
125°F	0.00125	0.00118	0.00089	0.00169	
275°F	-	0.00125	0.00084**	0.00185	0.
<u>Thermal Conductivity,* Virgin Material - Heat Soaked 5 Min at 400°F (Btu/in./hr - ft<sup>2</sup>-°F)</u>					
RT	1.455	1.260	1.265	1.434	1.
125°F	0.810	1.395	1.085	1.640	0.
200°F	0.985	1.090	1.126	1.425	1.
275°F	0.920**	1.530**	0.820**	1.240	0.
<u>Thermal Conductivity, Virgin Material (Btu-in/hr-ft<sup>2</sup>-°F)</u>					
125°F	1.760	1.510	1.255	2.250	
275°F	-	1.750	1.120**	2.640	1.

\* "Apparent" thermal conductivity of heat soaked material (heat capacity component was not fired for  
 \*\* At 250°F

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<u>V50</u>	<u>V51</u>	<u>V61</u>	<u>SD 850-15C</u>	<u>40SA2</u>	<u>SMR 81-15</u>	<u>USR 3804</u>
0.00113	0.00089	0.00066	0.00105	0.00109	0.00106	0.00118
0.00123	0.00078	0.00079	0.00102	0.00105	0.00129	0.00126
0.00104	0.00083	0.00063	0.00113	0.00100	0.00106	0.00105
0.00087	0.0005**	0.00076	0.00093	0.00094	0.00106	0.00093
0.00169	-	0.00121	0.00118	0.00143	0.00143	0.00112
0.00185	0.00150	0.00088	0.00110	0.00114	0.00155	0.00151
<u>2 OF</u>						
1.434	1.113	0.795	1.330	1.390	1.185	1.460
1.640	0.996	1.030	1.345	1.410	1.555	1.610
1.425	1.085	0.867	1.520	1.390	1.390	1.410
1.240	0.655**	1.080	1.255	1.330	1.470	1.260
2.250	-	1.560	1.555	1.920	1.725	1.430
2.640	1.955	1.260	1.485	1.615	2.150	2.060

was not fired from heat soak material).

Figure 10. Significant Properties of Ten Remaining Materials

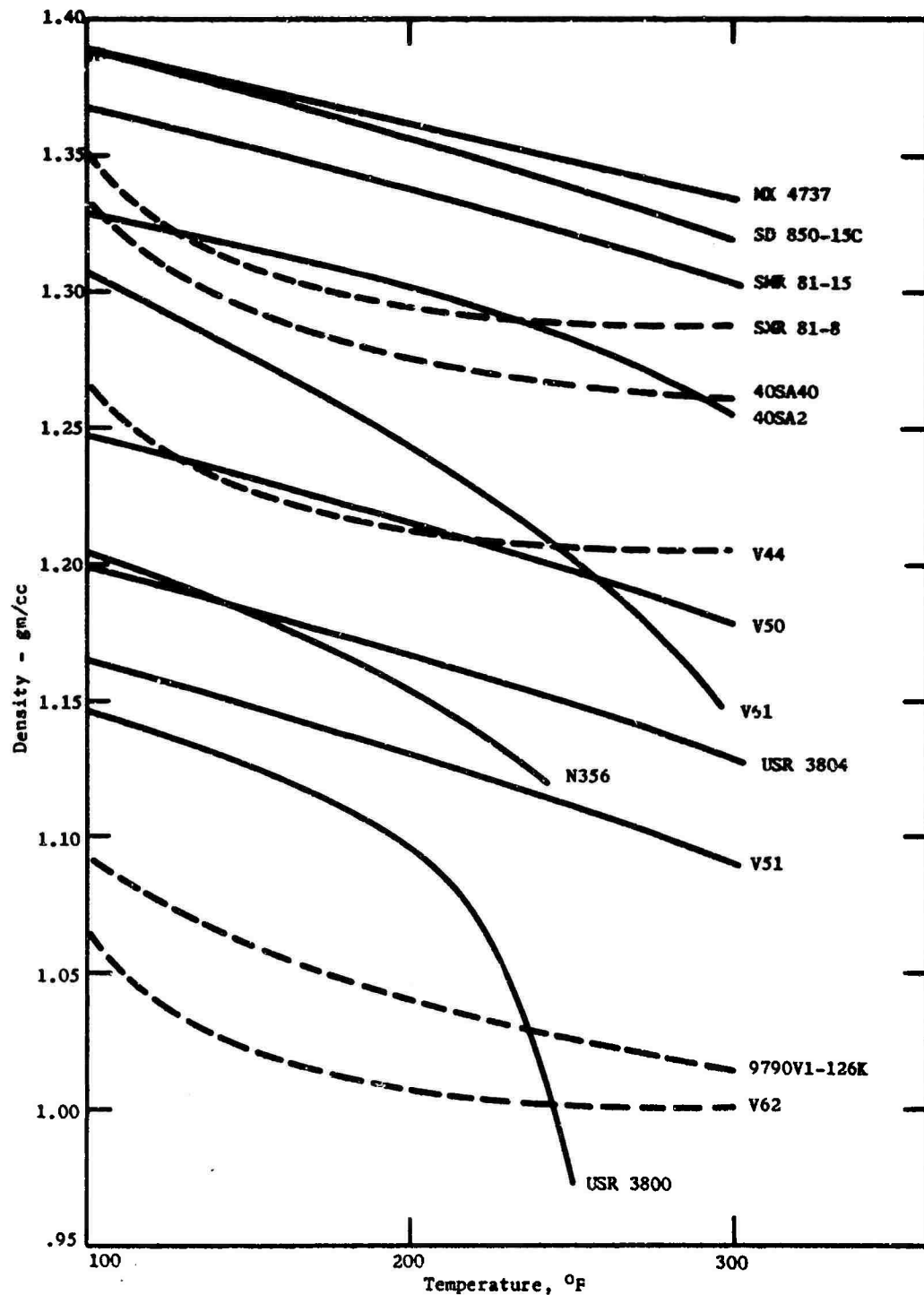
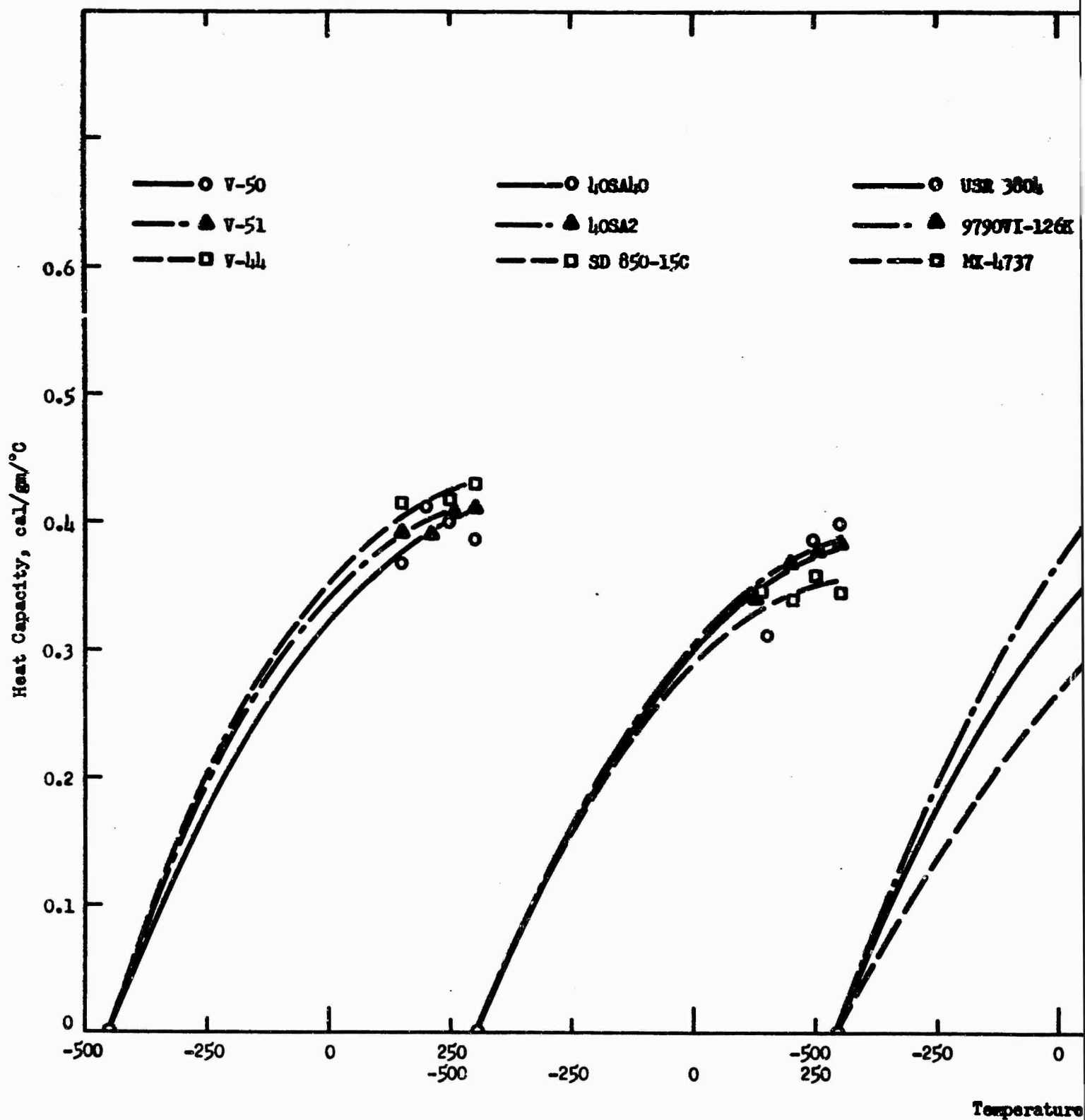


Figure 11. Density of Virgin Materials





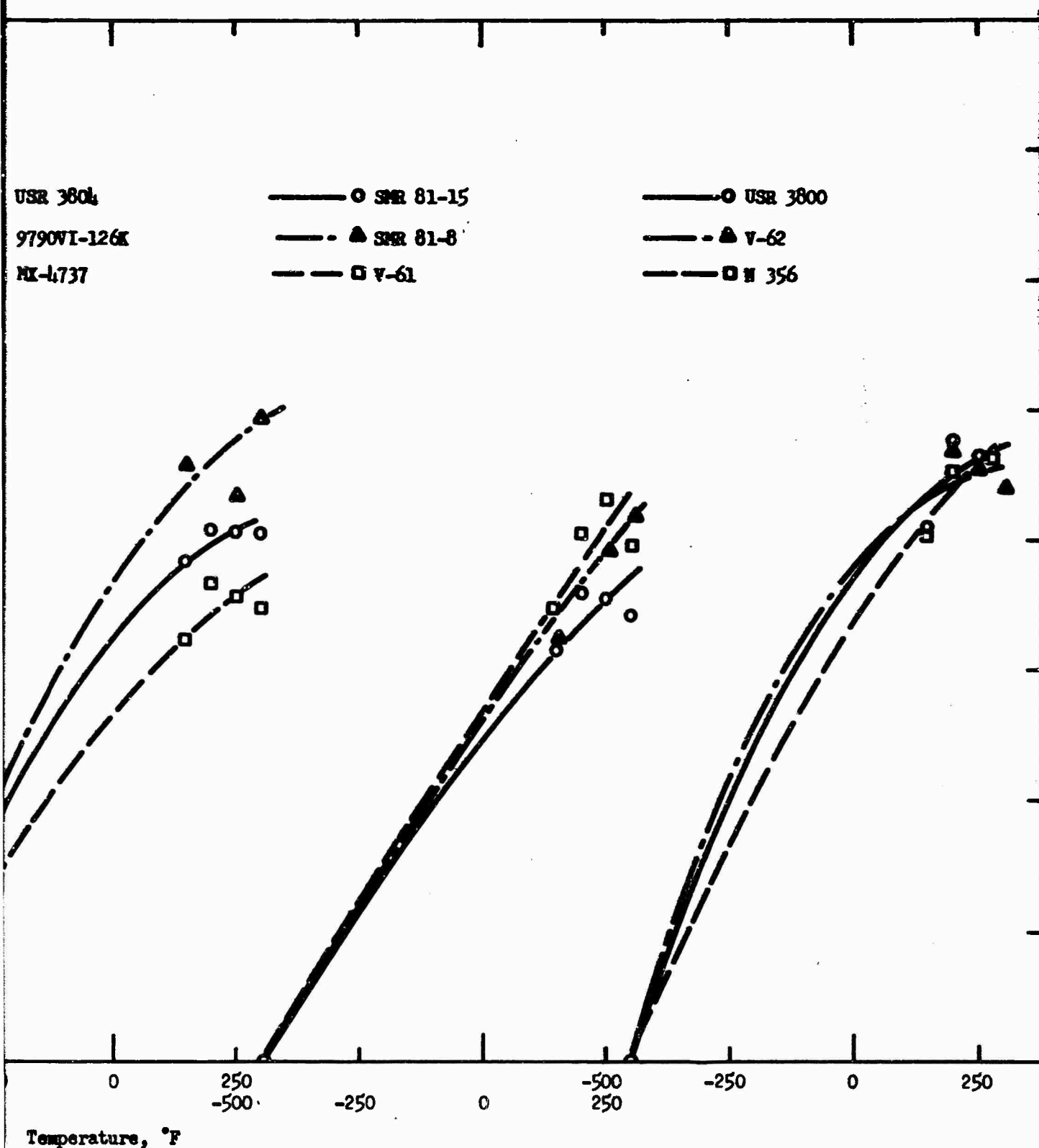
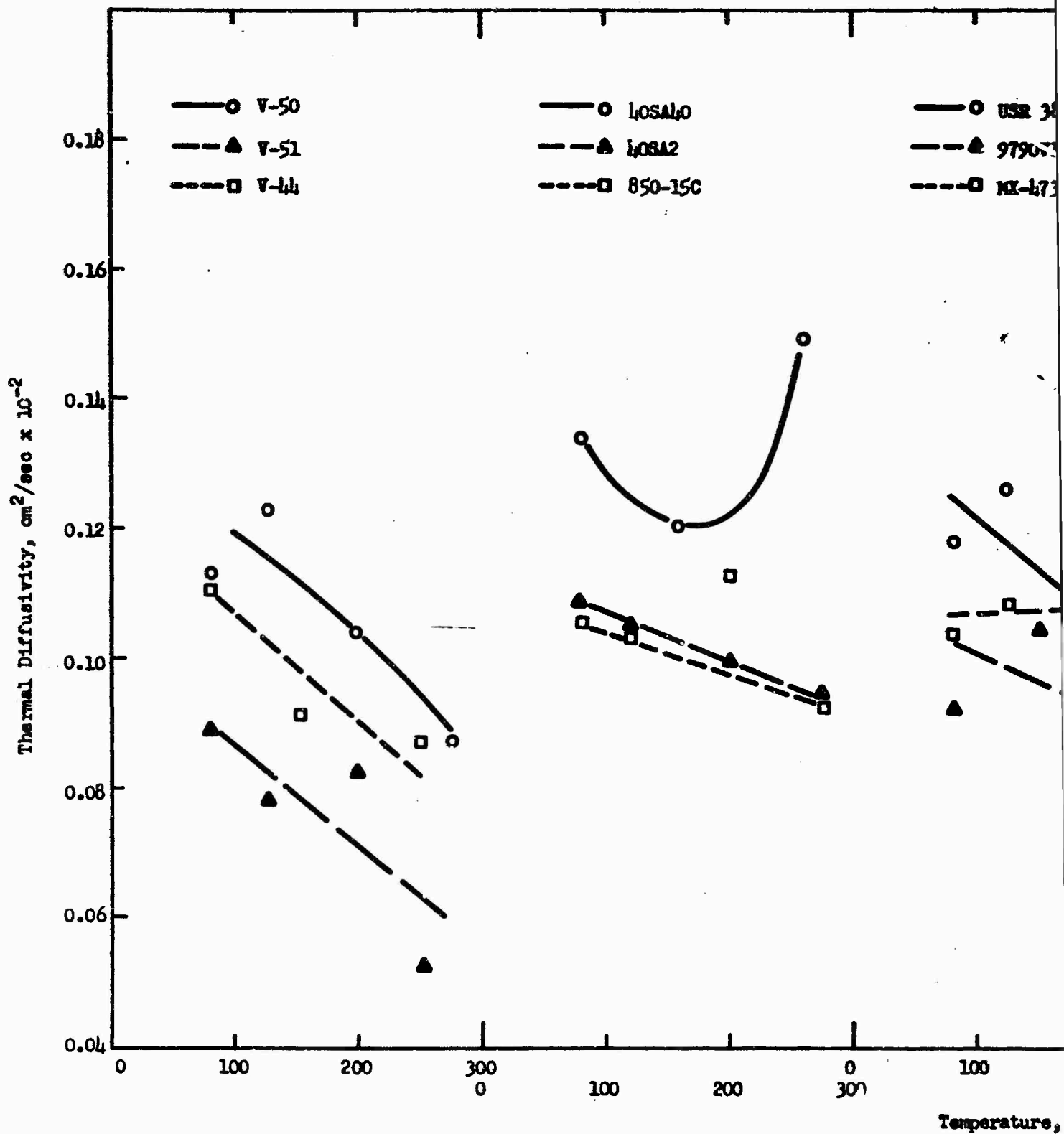
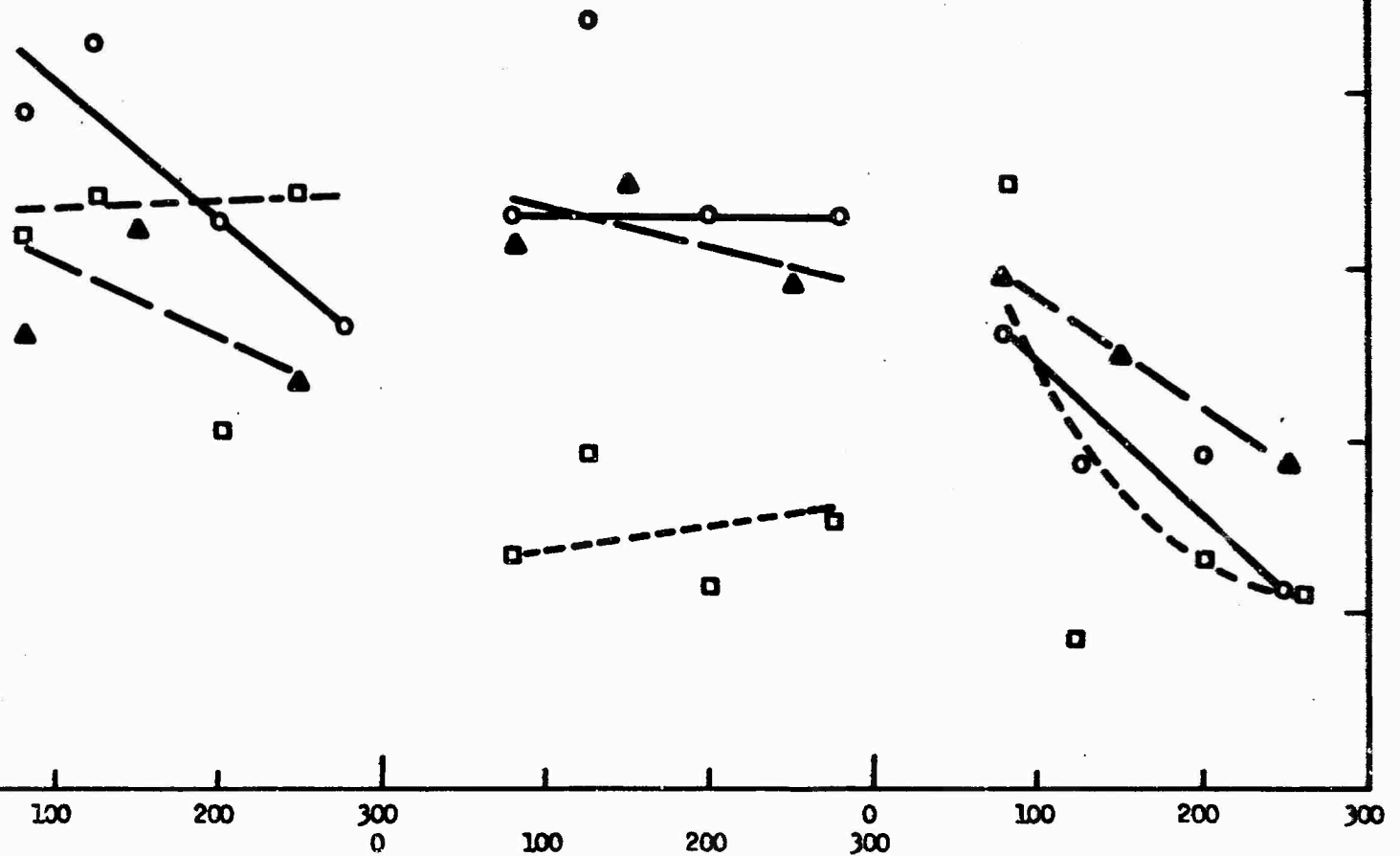


Figure 12. Heat Capacity of Virgin Materials

2



—○— USR 3804	—○— SRR 81-150	—○— USR 3800
—△— 9790VI-126K	—△— SRR 81-8	—△— V-62
—□— MX-4737	—□— V-61	—□— N 356



Temperature, °F

Figure 13. Thermal Diffusivity of Virgin Materials  
(Heat Soaked for Five Minutes at 400°F)

2

III, B, Testing for Significant Properties (Task D, Phase I) (cont.)

5. Thermal Conductivity

Thermal conductivity data were calculated from the density, heat capacity, and diffusivity results as follows:

$$K = \alpha \rho C_p$$

where

$$K = \text{thermal conductivity, Btu-in./hr-ft}^2\text{-}^\circ\text{F}$$

$$\alpha = \text{thermal diffusivity, ft}^2\text{/hr}$$

$$\rho = \text{density, lb/ft}^3$$

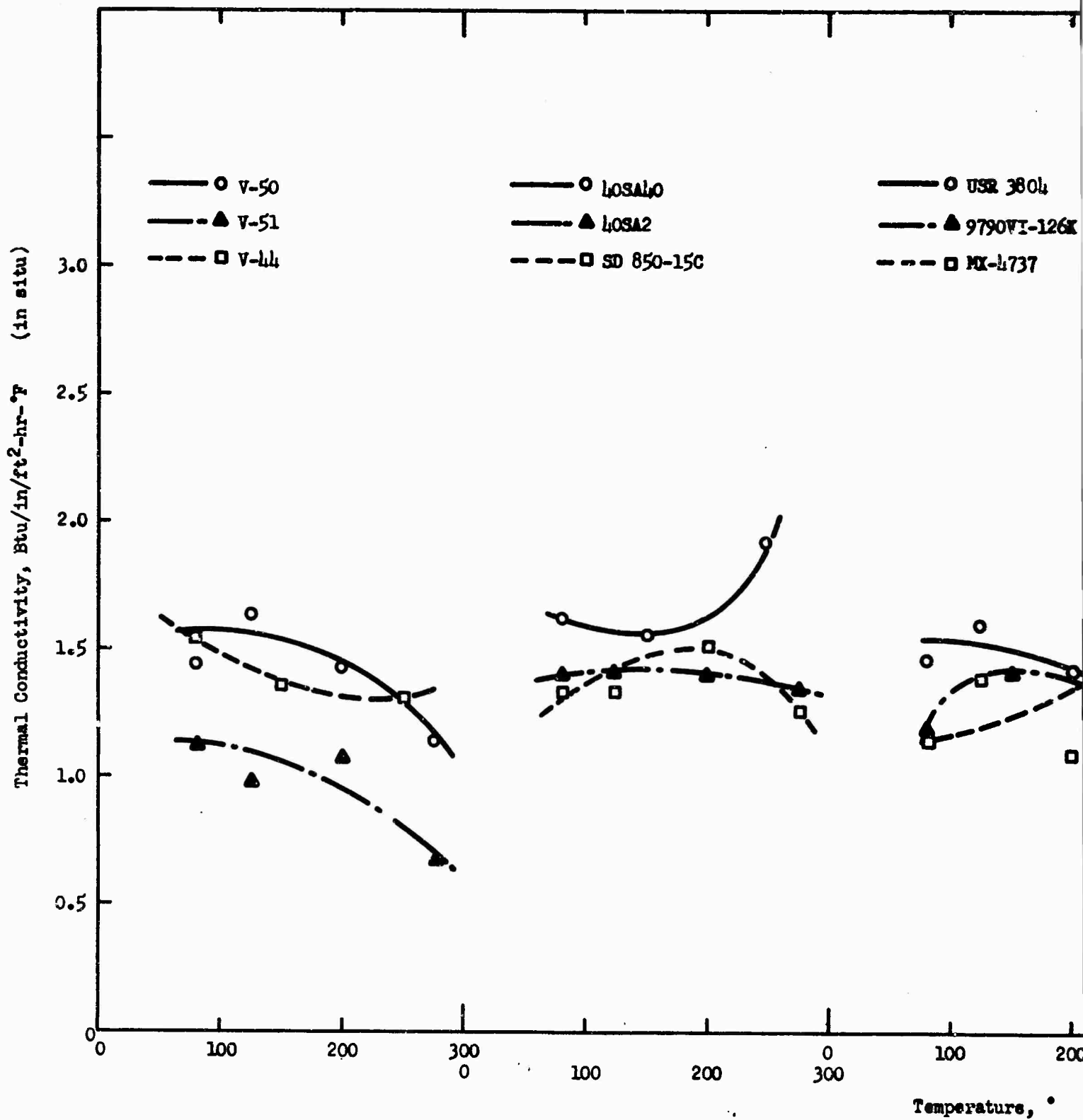
$$C_p = \text{specific heat, Btu/lb/}^\circ\text{F}$$

The specific heats and density values were at the same temperature conditions as the thermal diffusivities.

Results of the calculations are shown in Figure 10. Complete data were obtained on an "apparent" type thermal conductivity for heat-soaked virgin materials, "apparent" because while the diffusivity values were for heat soaked materials, the heat capacity values were for virgin materials. Heat capacities of the virgin materials were originally obtained because they were shown to be significant in the correlation analysis. Additional spot checks are currently being conducted on heat-soaked materials to show the extent of change resulting from heat soak. Partial calculations were also made on the thermal conductivity of virgin materials for comparative purposes. Results of the "apparent" calculations are presented in Figure 14 along with corresponding data on the original five materials. These data show that U61, USR3800, U62, N356, and U51 have the lowest thermal conductivities.

6. Heat-Soak Tests

During preparation of the heat soaked specimens for the diffusivity tests, it was noted that certain of the materials exhibited considerable permanent volumetric changes. Typical examples of the swelling are shown in Figures 15 and 16 for all 15 materials. The worst of these materials was N356. As the heat soaking was conducted at ambient pressure, the results are not indicative of what happens at elevated pressures during motor firing, but would indicate what might occur during shutdown between pulses.



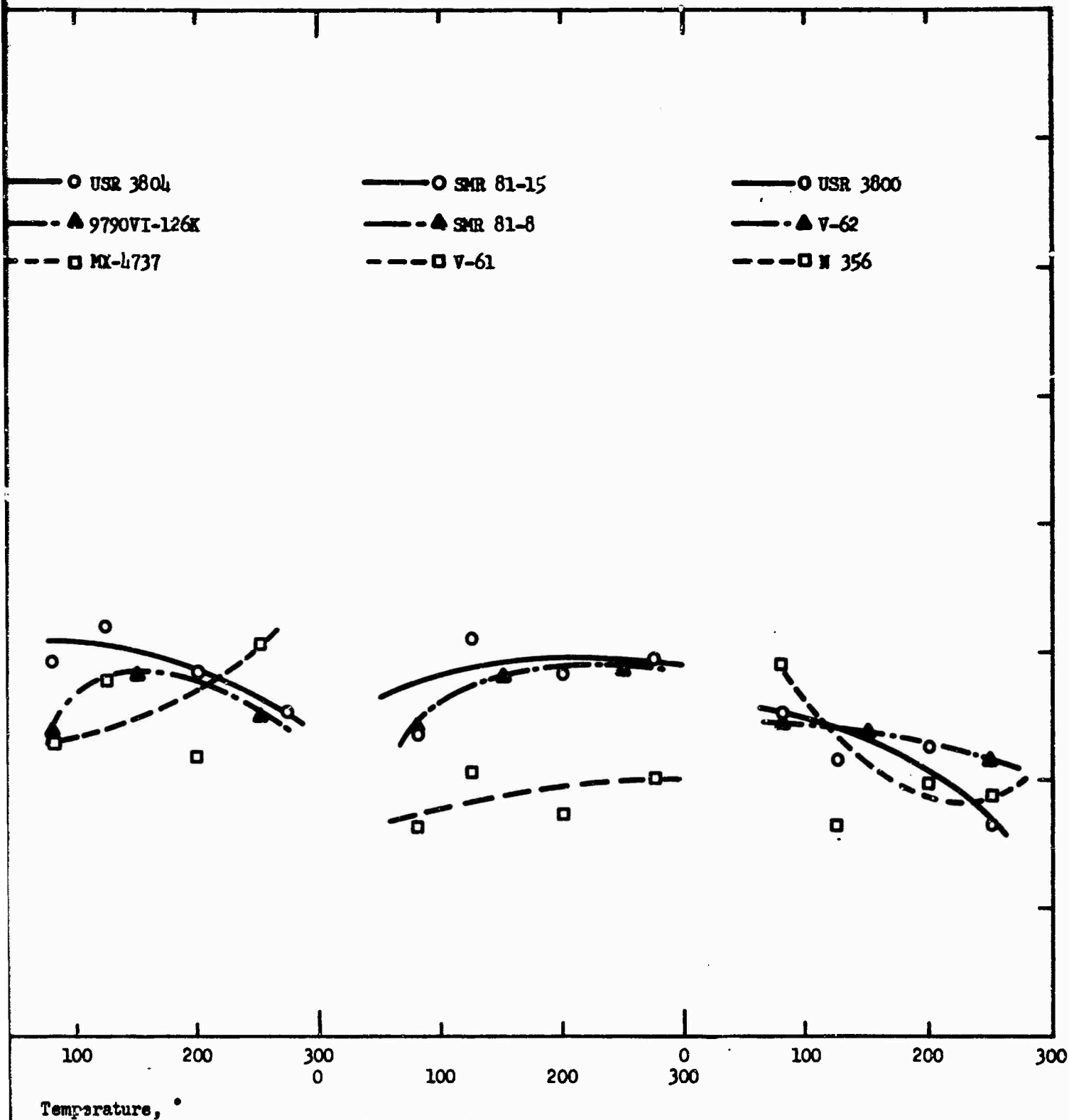


Figure 14. "Apparent" Thermal Conductivity of Virgin Materials  
(Heat Soaked for Five Minutes at 400°F)

2

ORIGINAL SAMPLE SIZE : 2" x 1/2" x 1/2"











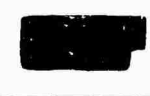
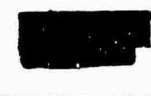



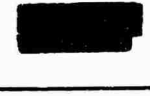

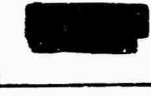
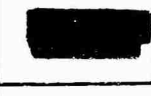
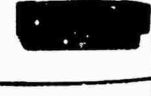
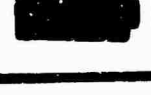
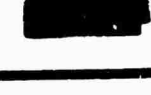
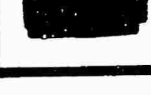
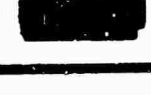

	500°F			400°F	300°F
	30 min	10 min	5 min	30 min	30 min
SMR 81-8 (BUTYL)					
40SA40 (POLYURETHANE)					
9790VI-126A (EPR)					
GEN-GARD V44 (NBR)					
V62 (SBR-PHENOLIC)					

Figure 15. Volume Change on Heat Soak at 500° and 400°F

ORIGINAL SAMPLE SIZE : 2" x 1/2" x 1/2"















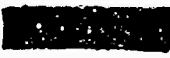





MATERIAL	ORIGINAL	HEAT SOAKED 5 min AT 400°F
40SA-2 (POLYURETHANE)		
V-61 (EPOXY-POLYSULFIDE)		
MX 4737 (NBR PHENOLIC)		
V 50 (NBR)		
V51 (NBR)		
850-15C (PBAN-EPOXY)		
USR 3904 (EPR)		
SMR 81-15 (BUTYL)		
USR 3800 (NBR PHENOLIC)		
N-356 (NBR PHENOLIC)		

Figure 16. Volume Change on Heat Soak at 400°F



### III, Technical Discussion (cont.)

#### C. SELECTION OF BEST MATERIALS (TASK F, PHASE I)

Materials for use in Phase III of the program were selected by (1) utilizing the significant property data for the 15 candidate materials together with plasma-arc test data as a screening mechanism for selecting the best candidates from an ablation standpoint and by (2) reviewing other material characteristics such as processability, service life, storage stability, and demonstrated capability. A discussion of each category follows.

##### 1. Selection for Ablative Performance

In arriving at the best materials for ablation, the heat capacity, diffusivity, and heat of combustion properties of the candidate materials were first reviewed. Each material was given a rating of from 1 to 15 for each of these properties, as shown in Figure 17 with 1 being the highest rating. (Thermal conductivity was not included in the subrating because this would be duplicating heat capacity and diffusivity weight factors.) A subtotal was then established on the basis of this review.

The property values and plasma arc data for each of the candidate materials were then used as input to the screening equations derived in Task C, Phase I, to estimate the ablation rates that would be expected if the materials were used in an environment similar to that encountered in the three-pulse motor. Results obtained from these equations are also shown in Figure 17. The overall rating indicates that, insofar as ablation performance is concerned, the best material would be USR 3800 followed by V62, N356, 9790V1-126K, V51, V44, and USR 3804.

##### 2. Selection Based on Material Characteristics

To aid in the selection of the best materials, an evaluation of all 15 candidate materials was made on the basis of processability, general mechanical and thermal properties, adhesive properties, storage stability, demonstrated capability in motors, and raw material costs. A rating system for each material based on all characteristics is shown in Figure 18. In this rating system, processability was arbitrarily given a maximum weight of 15 points; general mechanical and thermal properties and adhesive properties each 10 points; and demonstrated capability, storage stability, and raw material cost each 5 points. On the basis of the material characteristics, the 40SA40 ranks as the best material followed by the EPR materials, 9790V1-126K and USR 3804. A discussion of each of the characteristics is given below:

##### a. Processability

The assessment of the processability of the 15 materials was made on the basis of in-house and supplier data. Factors that were evaluated include ease of processing, bondability, processing cost, documentation, and ease of repair (see Figure 19).

<u>Material</u>	<u>Significant Properties Rating</u>				<u>Plas Equ</u>
	<u>Heat Cap @ 250°F</u>	<u>Diffusivity @ 250°F</u>	<u>Heat of Comb</u>	<u>Sub Total Rating</u>	
V62	4	5	1	3	
N356	3	1	6	3	
UBR 3800	2	1	5	1	
UBR 3804	8	11	3	8	1
9790V1-126K	1	6	2	2	1
V44	5	6	8	7	1
V51	7	1	4	5	1
SD 850-15C	15	8	7	10	1
MX 4737	13	14	15	15	1
V50	9	8	9	9	1
V61	6	4	10	6	1
SMK 81-8	10	12	11	11	1
SMR 81-15	14	13	14	14	1
408A40	12	15	13	13	1
408A2	11	10	12	11	1

Expected Ablation Rate- Three Pulse Motor @ 225 Btu/ft <sup>2</sup> -sec			
<u>Plasma Arc Equation</u>	<u>Two Variable Equation</u>	<u>Subtotal Rating</u>	<u>Performance Rating</u>
9.7	9.8	1	2
9.7	10.0	2	3
9.8	9.7	1	1
10.3	10.8	7	7
10.4	10.2	5	4
10.5	10.6	6	6
10.5	9.7	4	5
10.5	10.9	8	9
10.6	12.2	11	13
10.6	11.2	10	10
11.0	10.6	9	7
11.3	11.5	11	11
11.4	11.9	13	14
12.9	12.9	15	15
13.6	11.6	14	12

Figure 17. Rating of 15 Candidate Materials for Ablation Performance

2

I. Material Characteristics	Significance	V62	N-356	USR 3800	9790V1- 126K	USR 3804	V44	V5
	Weight, Maximum							
<u>Processability</u>	<u>15</u>							
a. Ease of Processing	4	4	2	2	4	4	4	4
b. Bonding Operations	4	2	2	2	2	2	2	2
c. Processing Cost	3	1	1	1	1	1	1	1
d. Documentation & Specs	2	2	1	2	1	1	2	1
e. Ease of Repair	2	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>
Subtotal		10	7	8	9	9	10	9
<u>Mechanical Properties</u>	<u>10</u>							
a. Tensile/Elongation/ Modulus Properties	3	3	3	3	3	3	3	3
b. Low Temp. Properties	3	2	1	2	3	3	1	2
c. Service Temp. Range	4	<u>3</u>	<u>1</u>	<u>1</u>	<u>4</u>	<u>4</u>	<u>2</u>	<u>3</u>
Subtotal		8	5	6	10	10	6	8
<u>Adhesive Properties</u>	<u>10</u>	6	7	7	8	8	9	7
<u>Demonstrated Performance Capability</u>	<u>5</u>	3	2	4	3	3	5	2
<u>Storage Stability</u>	<u>5</u>	3	3	3	5	5	3	3
<u>Material Cost</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>
Total - Points	50	35	29	33	40	40	37	34
Characteristics Rating		10	14	13	2	2	7	11
II. Ablative Performance Rating		2	3	1	4	7	6	10
Final Overall Rating		3	11	6	1	2	4	13

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<u>SR 3804</u>	<u>V44</u>	<u>V50</u>	<u>V51</u>	<u>SD850 -15C</u>	<u>MX4737</u>	<u>V61</u>	<u>SMR 81-8</u>	<u>SMR 81-15</u>	<u>40 SA40</u>	<u>40 SA2</u>
4	4	4	4	2	3	2	4	4	3	2
2	2	2	2	4	2	4	1	1	4	4
1	1	1	1	3	1	3	1	1	3	2
1	2	1	1	1	1	2	2	2	2	1
<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>2</u>	<u>1</u>	<u>2</u>	<u>1</u>	<u>1</u>	<u>2</u>	<u>2</u>
9	10	9	9	12	8	13	9	9	14	11
3	3	3	3	2	2	2	3	3	3	3
3	1	2	2	1	1	1	3	3	3	3
<u>4</u>	<u>2</u>	<u>3</u>	<u>3</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>4</u>	<u>4</u>	<u>4</u>	<u>4</u>
10	6	8	8	4	4	4	10	10	10	10
8	9	7	7	10	7	8	5	5	10	10
3	5	2	2	4	3	4	4	4	4	2
5	3	3	3	3	3	3	4	4	3	3
<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>3</u>	<u>5</u>	<u>5</u>	<u>5</u>	<u>3</u>	<u>3</u>
40	37	34	34	38	28	37	37	37	44	39
2	7	11	11	5	15	7	7	7	1	4
7	6	10	5	9	13	7	11	14	15	12
2	4	13	9	6	15	6	12	13	9	9

Figure 18. Overall Rating of 15 Materials

2

<u>Material</u> <u>Class (Binder)</u> <u>Code</u>	<u>Available</u> <u>Forms</u>		<u>Processing and</u> <u>Installation</u> <u>Methods</u>				<u>Specific</u> <u>Processing</u> <u>Problems</u>	<u>Ex</u> <u>Proc</u> <u>Inst</u>
	Calendered Sheet Stock	Two-Liquid Components	Premolding and Secondary Bonding Pre-Cast (Trowel) & Cure/Sec. Bonding	Lay-Up	In-Place Troweling	In-Place Casting	Tendency to form voids on installation Tendency to form voids on cure	Premolding (Trowel, Cast) & Sec. Bonding
<u>SR-Phenolic</u>								
V62	X		X	X				Good
<u>NR-Phenolic</u>								
N-356	X		X	X			X	Good
USR 3800	X		X	X			X	Good
MX 4737	X		X	X				Good
<u>EPR</u>								
9790V1-126K	X		X	X				Good
USR 3804	X		X	X				Good
<u>NR</u>								
V44	X		X	X				Good
V50	X		X	X				Good
V51	X		X	X				Good
<u>PBAN-Epoxy</u>								
SD850-15-C		X	X		X		X	Fair
<u>Polysulfide-Epoxy</u>								
V-61		X	X		X		X	Fair
<u>Butyl</u>								
SMR 81-8	X		X	X				Good
SMR 81-15	X		X	X				Good
<u>URETHANE</u>								
40 SA-40		X	X			X	X	Good
40 SA-2		X	X			X	X	Fair

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Ease of Processing and Installation			Bonding			Processing Cost	Documented Processing and Installation Methods					Ease of Repair		
Premolding (Trowel., Cast) & Sec. Bonding	Lay-Up	Troweling	Casting	Bonds to Chamber Without Primer and Adhesive	Bonds to Propellant Without Use of Liner	Bonding to Chamber With Adhesive/Primer	Bonding to Liner (Propellant)	Processing	AGC Spec	Vendor Spec	MIL/WS Spec		AGC Procedure	Vendor Procedure
Good	Good				Good	Fair		High	X	X			X	Fair
Good	Poor				Good	Fair		High		X			X	Fair
Good	Poor				Good	Fair		High		X	X		X	Fair
Good	Fair				Good	Fair		High		X			X	Fair
Good	Good				Good	Fair		High		X			X	Fair
Good	Good				Good	Fair		High		X			X	Fair
Good	Good				Good	Fair		High	X	X	X	X	X	Fair
Good	Good				Good	Fair		High		X		X		Fair
Good	Good				Good	Fair		High		X		X		Fair
Fair	Fair			X	X	Ex. Good		Low	X			X		Good
Fair	Fair			X		Ex. Good		Low	X	X	X			Good
Good	Good				Fair	Fair		High	X	X	X			Fair
Good	Good				Fair	Fair		High	X	X	X			Fair
Good		Fair		X	X	Good	Good	Low	X		X	X	X	Good
Fair		Fair		X	X	Good	Good	Low					X	Good

Figure 19. Comparison of 15 Materials--Processing and Installation

2

III, C, Selection of Best Materials (Task F, Phase I) (cont.)

The 15 materials are available either in the form of calendered sheet stock or as a two-liquid component system. As indicated in Figure 19, each material can be used for prefabrication of an insulation part with the resulting part installed in the chamber by secondary bonding, or each material can be installed in the chamber prior to cure by lay-up, troweling, or casting. The laid-up rubber insulation is cured in an autoclave at 100 to 200 p.s.i. pressure at about 300°F. The trowelable and the castable materials are cured at normal atmosphere and at low temperatures such as 180°F for the urethanes, 135°F for the SD 850-15C, and ambient for V61. The premolding method, which requires rather costly molds of stainless steel, is generally used for high part production; vice-versa, the lay-up method is used when a small number of chambers are to be insulated.

V62 materials of the SBR-phenolic class can easily be processed by premolding or lay-up. The NBR-phenolic materials N-356, USR 3800 and MX 4737 can also be processed and installed similar to the SBR-phenolic material. However, both the N356 and the USR 3800 materials contain fillers which decompose below 300°F. To avoid expansion during cure, these materials should be contained in steel molds. The lay-up method, therefore, is not generally suitable for N356 and USR 3800.

The new EPR materials, 9790V1-126K and USR 3800, have exhibited good processability both in the premolding and lay-up methods. The processability of V44 has been evaluated extensively in many rocket motor programs such as Polaris and Minuteman and found to be good both by the premolding and lay-up procedures. V50 and V51 are modifications of V44 and estimated to be similar to V44 in processability. The V44 forms a good bond to some polyurethane propellants but a liner between the propellant and insulation is usually required to provide good bonding to these materials.

The PBAN-epoxy material SD 850-15C has epoxy resin as a main ingredient and does not contain any plasticizer. One problem in processing this material by troweling is void formation because of high viscosity of the material. The polysulfide-epoxy material V61 is similar to SD 850-15C in processing characteristics. Its processing and installation methods have been well documented because it is widely used as a repair material. The urethane materials 4OSA40 and 4OSA2 are quite viscous as castable materials. Voids in the cast parts sometimes occur, particularly in parts of 4OSA2.

The butyl materials SMR 81-8 and SMR 81-15 are similar in composition and ease of processability. Some difficulties have been encountered in obtaining good bond between vulcanized parts of these materials. The bonding operation requires caution in cleaning and in applying primer and adhesive.



III, C, Selection of Best Materials (Task F, Phase I) (cont.)

b. General Mechanical and Thermal Properties

An evaluation of the 15 materials was also made on the basis of their applicability for motor handling, temperature cycling, storage, and rapid pressurization of the motors. The evaluation was based partly on determinations made in this and other programs and partly on the basis of suppliers data. The data in Figure 20 show that the EPR materials 9790V1-126K and USR 3804, the Butyl materials SMR 81-8 and SMR 81-15, and the polyurethane materials 40SA40 and 40SA2 have good mechanical properties for use over a temperature range from -75 to 300°F. The NBR materials V50 and V51 have an estimated service temperature range from -45 to 300°F. V62 also exhibits good mechanical properties over a wide range, -40 to 300°F. The other materials apparently should not be used at temperatures below 0°F as indicated by the temperatures at which they become brittle.

c. Adhesive Properties

In stop-start motors with end-burning grains similar to those used during this program, the insulation is bonded only to the chamber wall. No boots are used and the restricted propellant grains are not bonded to the insulated chamber. In other configurations, however, case bonded propellants are used. The assessment of the adhesive properties of the 15 candidate materials was made only on the basis of bonding the insulation to the chamber wall. Bonding of insulation to propellant was not considered since this is so greatly dependent on the type of propellant used.

The requirements imposed on the insulation-chamber bond is that it must resist failure under the environments of heat soak during shutdown, temperature cycling, storage, handling, and pressurization cycling. A review of bond data obtained in rocket programs and supplied by vendors indicates that all of the 15 materials should have sufficient bond strength to metal and Fiberglas chambers for stop-start motor applications. Bond data previously reported in this program for the five representative materials (V44, V62, 40SA40, 81-8, and 9790V1-126K) showed no detrimental changes after cycling 10 times between -65 and 250°F (Figure 21). After heat soak at 250°F for 5 and 30 minutes, all five materials except 40SA40 attained increased bond strength. Data showing effects of heat soak and temperature cycling are not available for the other ten materials. Estimates of the bondability in stop-start applications, however, can be made on the basis of standard tensile and lap-shear values, thermal stability data and in some cases, on the basis of a close relationship in formulations to the five materials evaluated. For example, the SMR 81-15 differs from 81-8 only in filler composition and the 40SA2 differs from 40SA40 only in the ratio of binder ingredients. Also, the USR 3804 material is similar to the EPR 9790V1-126K material in composition. Estimated minimum bond data, therefore, are given in Figure 21 to facilitate a comparison.

Material	Tensile Strength psi	Elongation % @ 77°F	Mod. (E) psi	Elongation @ -65°F, %	Brittle Point <sup>(1)</sup> °F	Torsional Stiffness <sup>(2)</sup> psi °F	Estimated Service Temp. Range, °F
V62	675/740/1789			> 40	-44	5,000 -40 40,000 -54	-40 to 300
M-B56	1200/200/---			--	20	--	30 to 300
USR 3800	1200/325/---			2	10	--	15 to 300
MX 4737	1200/14/---			--	10	--	15 to 300
9790VI-126K	2003/797/5300			> 30	-80	--	-75 to 300
USR 3804	1200/400/---			> 30	-80	--	-75 to 300
V44	1446/467/1378			2-3	-6	5,000 9 40,000 -15	0 to 300
V-50	1200/400/1300			> 40	-49	5,000 -29 40,000 -52	-45 to 300
V-51	2500/600/---			> 40	-53	5,000 -36 40,000 -54	-45 to 300
SD850-15C	945/105/3500			2	30	--	30 to 300
V61	900/(2-5)/---			2	30	--	30 to 300
SMR 81-8	934/350/2200			> 30	-80	5,000 -57 40,000 -81	-75 to 300
SMR 81-15	Similar to 81-8			> 30	-80	5,000 -54 40,000 -81	-75 to 300
40 SA 40	1126/133/526			> 30	-80	5,000 63 40,000 -49	-75 to 300
40 SA 2	Similar to 40 SA 40			> 30	-80	--	-75 to 300

(1) ASTM D 746-57T (Suppliers data)

(2) ASTM D 1043-61T (Apparent Modulus of Rigidity), suppliers data

Figure 20. Comparison of 15 Materials--Mechanical Properties

<u>Adhesive Properties</u>	<u>444</u>	<u>9790V1 126K</u>	<u>V62</u>	<u>81-8</u>	<u>81-15</u>	<u>408A40</u>	<u>40</u>
<u>Bonding to Chamber Materials</u> (4130 Sheet)							
Tensile Bond Strength @ 77°F, psi	408	> 300	> 300	> 300	408	1325	> 1
Lap Shear Strength, psi							
Original @ 77°F	836	449	148.6	113.4	> 150	496	>
Heat Soaked at 250°F	705	350	160.6	115.2		180	>
Original @ -65°F	488	2146	192	156		1735	> 1
Ten Cycles, -65 to 250°F	478	1570	722.0	257		1482	> 1
<u>Demonstrated Performance</u> <u>Capability in Rocket Motor</u>							
Polaris	X						
Polaris Test Motor	X						
Minuteman	X		X				
Large Booster Motors	X						
Upper-Stage Motor			X				
Rebel & Phoenix				X		X	
Sparrow					X		
DM14 - Multiple Start	X	X	X	X		X	
AGM				X		X	
Sub-Scale Test Motors			X				
<u>Storage Stability</u>							
Resistance to O zone	Fair	Excellent	Fair	Excellent	Excellent	Good	Go
Resistance to Ultraviolet Light	Good	Excellent	Good	Excellent	Excellent	Good	Go
Estimated Service Life in Rocket Motors of Ambient Conditions, Years	5-10	> 15	5-10	10-15	10-15	5-10	5-
<u>Raw Material Cost \$/lb</u>							
< 5.00	X	X	X	X	X		
> 5.00						X	X

	<u>408A40</u>	<u>408A2</u>	<u>N-356</u>	<u>3800</u>	<u>MX4737</u>	<u>3804</u>	<u>V50</u>	<u>V51</u>	<u>SD850-150</u>	<u>V61</u>
08	1325	> 1300	> 300	> 300	> 300	> 300	356	> 300	531	> 400
50	496 180 1735 1482	> 500 > 200 > 1800 > 1400	> 200	> 200	> 200	> 200	> 400	> 400	684	> 400
						X X			X	X  X
	X  X X								X	X
Excellent Excellent	Good Good	Good Good	Good Good	Good Good	Good Good	Excellent Excellent	Fair Good	Fair Good	Good Good	Good Good
0-15	5-10	5-10	5-10	5-10	5-10	> 15	5-10	5-10	5-10	5-10
			X			X	X	X	X	X
	X	X			X					

Figure 21. Comparison of 15 Materials--Adhesive Properties, Demonstrated Capability, Storage Stability, and Raw Material Cost

2

### III, C, Selection of Best Materials (Task F, Phase I) (cont.)

The bond data show that the epoxy-cured PBAN and polysulfide materials (SD 850-15C and V61) and the polyurethane materials (40SA40 and 40SA2) have excellent bondability. For these materials, no primers are required when they are trowelled or cast into position. High bond-strength values also have been exhibited by the NBR materials when applied both as green stock during lay-up and as vulcanized rubber. The NBR-phenolic materials, in general, have quite high bond strength to metal and fiberglass substrates. The SBR material, V62, had one of the lower bond strengths. An evaluation of the bondability of insulation materials in the Minuteman program also showed that V62 had lower bond values than V44. The butyl rubber materials SMR 81-8 and SMR 81-15 exhibit the lowest bond strength values. Problems which have been encountered in the bonding of these butyl materials generally have been caused by the use of an improper primer or adhesive or to improper application methods for the primer or adhesive.

#### d. Storage Stability, Demonstrated Performance Capability, and Material Cost

A comparison of estimated storage stability in years of service life, demonstrated performance capability, and raw material costs for the 15 materials is included in Figure 21. The estimated service life is based on data reported in technical literature, in military and commercial applications, on rocket motor aging data, and on the resistance of materials to degradation on exposure to heat, light, and oxone. The comparison shows that the butyl and EPR materials have excellent storage stability. All of the other materials are estimated to have a service life from five to ten years in motors at ambient conditions (materials stored in a relaxed condition and not exposed to ultraviolet light). Figure 21 indicates that 9 of the 15 materials have demonstrated good performance in rocket motors.

A comparison of raw material cost based on price per pound above and below \$5.00 for the materials also is included in Figure 21. This cost applies to the materials when supplied as calendered sheet or in the form of a two-liquid component system. The significance of the raw material cost is dependent on size and number of chambers to be insulated. For small motors, the ratio of material cost to installation cost is considerably lower than for larger motors such as Polaris and Minuteman.

### 3. Overall Selection of Best Materials

In selecting the best materials for use in the verification motors, the assumption was made that the general characteristics of the materials are relatively as important as ablative performance and an equal weight scale was set up as shown in Figure 18. Based on this rating system, the four best materials are expected to be from among 9790V1-126K, USR 3804, V62, V44, USR 3800, SD 850-15C and N356. N356 was selected because its good

III, C, Selection of Best Materials (Task F, Phase I) (cont.)

ablation properties should overshadow some of its other limitations. From these materials, four prime candidates (9790V1-126K, V62, V44 and USR 3800) and three secondary candidates (USR 3804, SD850-15C and N356) have been selected for installation in the 12- and 5-pulse verification motors. 40SA40 will be used as the "filler" material (see Figure 22). The prime specimens will be full-length specimens exposed to the complete range of area ratios available in the aft closure of the motors. The secondary specimens will be shorter and thus exposed to a more limited range of area ratios.

A brief summary of the capability of these materials follows:

a. Prime Candidates

9790V1-126K is an EPR material having excellent ablative properties, excellent operating temperature range ( $-75$  to  $300^{\circ}\text{F}$ ), and satisfactory bondability and processability.

V62 is an SBR-phenolic material having excellent ablative properties, good operating temperature range ( $-40^{\circ}$  to  $300^{\circ}\text{F}$ ), and satisfactory bondability and processability.

V44 is an NBR material with excellent ablative properties, nominal operating range ( $0$  -  $300^{\circ}\text{F}$ ), and excellent bondability and processability.

USR 3800 is an NBR-phenolic material with excellent ablative properties, nominal operating range ( $15$  -  $300^{\circ}\text{F}$ ), but only fair processability.

b. Secondary Candidates

USR 3804 is an EPR material similar to the 9790V1-126K material and presumably should be as good. The 9790V1-126K material was selected as a prime specimen rather than the USR 3804 because it was used in the correlation motors. Continued use of the 9790V1-126K in the verification motors will, thus provide additional data points.

SD 850-15C is a PBAN epoxy material having good ablative properties, nominal operating temperature range ( $30$  to  $300^{\circ}\text{F}$ ), and excellent bondability and processability.

N356 is an NBR-phenolic material having excellent ablative properties, nominal operating temperature ( $30$  -  $300^{\circ}\text{F}$ ), but only good processability. One characteristic of this material that might eliminate it from further consideration in pulse motors is the swelling noted during the heat-soak tests (see earlier discussion on this subject). This material will be closely observed during the static firing tests.

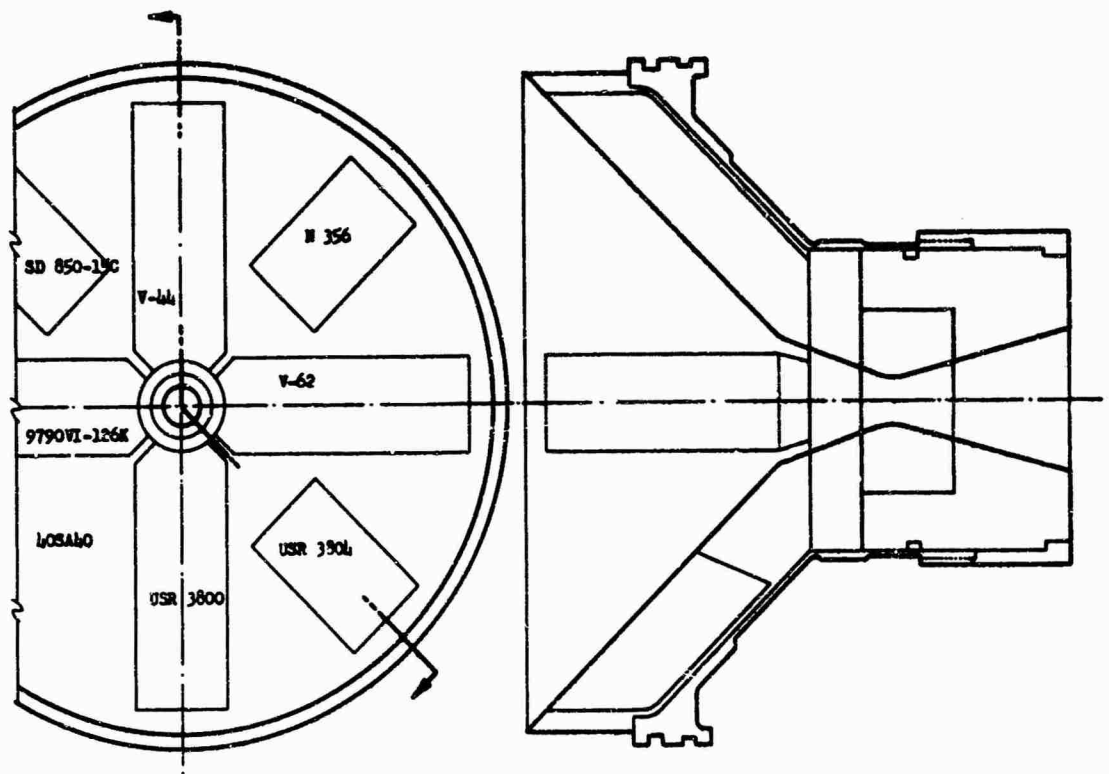


Figure 22. Location of Insulation Samples in 12- and 5-Pulse Motors

## III, C, Technical Discussion (cont.)

D. DETERMINE INFLUENCE OF INGREDIENTS ON PERFORMANCE AND PROPERTIES  
(TASK E, PHASE I)

The influence of ingredients on the performance and properties of elastomeric insulation materials in multiple restart applications was investigated by correlating ingredients against the ablation and regression performance and significant properties of five representative materials (V-44, V-62, 40SA40, SMR 81-8, and 9790V1-126K). The performances of these materials were obtained in a previous task in Phase I (Task B), where three successful motor firings were accomplished. The significant properties were those established in Task C (see paragraph A). The fillers, additives, and chemical ingredients used in the investigations were obtained for the virgin materials, char residues, and gases formed during pyrolysis. A partial listing of the ingredients is given in Figure 23. Complete identification of all ingredients is not included in this report because of the proprietary nature of the material formulations. Input to the correlation analysis consisted of the 74 variables shown in Figure 24. The confidence level was set at 95%, which resulted in a critical correlation coefficient of 0.88. Additional correlation coefficients down to the 90% confidence level are also of interest as an indication of a trend toward correlation. A positive correlation factor indicates an increase in ablation rate or regression rate as the ingredient factor increases and vice versa. Results of the analysis are discussed below:

1. Performance

The correlation coefficients for those components of the virgin materials, gas species, and char residues which have some significant relationship with one or more of the motor performance tests are shown in Figure 25. Although there are certain limitations in making a correlation analysis based only on five samples (see Appendix I), it appears from these data that a number of ingredients correlated with performance. As a previous correlation (Task C) indicated that there was little correlation between motor ablation and regression probably because of the loss of char between pulses, only those ingredients that correlated with ablation are discussed below:

## a. Weight % Carbon, Virgin Material

The carbon concentration of the five materials varied from 37 to 75% as shown in Figure 23. The correlation analysis indicates that as the concentration of the carbon increased, the ablation decreased. This relationship is further illustrated in Figure 26 for the three correlation motors at two heat flux levels--100 and 225 Btu/ft<sup>2</sup>-sec. The plots show that the best correlation of data was obtained at the 100 Btu/ft<sup>2</sup>-sec heat-flux level. This analysis suggests that a high carbon concentration in the insulation compound is significant for good performance.



<u>Ingredients</u>	<u>V62</u>	<u>9790V1-126K</u>	<u>V44</u>	<u>81-8</u>	<u>408A40</u>
<u>Binder</u>	SBR-Phenolic	EPR	NBR	Butyl	Urethane
<u>Main Fillers</u>	Asbestos Silica	Asbestos Silica	Asbestos Silica	Asbestos Silica	Silica TiO <sub>2</sub>
Wt% Inorganic Fillers	10.2	29.2	36.8	50.0	39.3
<u>Element Ratios, Virgin</u>					
C/O Ratio	8.3/1	4/1	2/1	1.3/1	1/1
C/H Ratio	9/1	6/1	10/1	6/1	6/1
O/H Ratio	1/1	1.5/1	4/1	5/1	5/1
<u>Weight %, Virgin</u>					
Wt% C	75	60	50	37	39
Wt% H	8	9	5	6	6
Wt% O	9	15	20	28	33
Wt% Si	3	11	12	18	9
Misc.	<u>5</u>	<u>5</u>	<u>13</u>	<u>11</u>	<u>13</u>
	100	100	100	100	100
<u>Decomposition Products</u>					
A. Gases at 550°C/350 psi					
Wt% CO	2.5	2.5	6.5	2.2	2.50
Wt% H <sub>2</sub> O	2.5	0.5	1.0	1.0	0.50
Wt% CO	3.5	2.5	6.5	1.0	9.50
Wt% H <sub>2</sub>	0.5	3.5	1.5	2.2	0.50
Wt% C <sub>n</sub> H <sub>n</sub> , Aliphatic	34.0	38.6	32.5	53.2	17.20
Wt% C <sub>n</sub> H <sub>n</sub> , Cyclic	2.7	1.0	1.9	0.4	5.7
Misc.	<u>0.3</u>	<u>1.4</u>	<u>0.1</u>	<u>-</u>	<u>-</u>
	46.0	50.0	50.0	60.0	35.5
B. Residue					
Wt% C	30.00	15.00	12.20	1.50	6.50
Wt% MgO	5.40	7.00	7.50	6.00	0.00
Wt% SiO <sub>2</sub>	12.00	20.00	20.00	25.00	29.00
Misc.	<u>6.60</u>	<u>8.00</u>	<u>10.30</u>	<u>7.50</u>	<u>29.50*</u>
	54.00	50.00	50.00	40.00	65.00
TOTAL A and B	100.00	100.00	100.00	100.00	100.00

Figure 23. Partial Listing of Ingredients

Virgin Material Ingredients

Weight % Polymer  
 Weight % Plasticizer  
 Weight % Filler  
 Weight % Aliphatic Carbons  
 C/O, C/H, O/H Ratio  
 Weight % C, H, O, N, S, Si, SiO<sub>2</sub>, H<sub>2</sub>O, and Asbestos

Properties of Composite Material

Density  
 Heat Capacity  
 Thermal Diffusivity  
 Heat of Combustion  
 Thermal Conductivity

Properties of Ingredients

Fusion (Softening) Temperature, Binder  
 Heat of Fusion, Fiber Filler  
 Heat of Fusion, Pigment Filler  
 Heat of Vaporization, Fiber Filler  
 Heat of Vaporization, Pigment Filler

Gaseous Decomposition Products

Weight % Total Gases  
 Weight % H<sub>2</sub>, H<sub>2</sub>O, CO, CO<sub>2</sub>  
 Weight % Total C<sub>n</sub>H<sub>n</sub>  
 Weight % Cyclic and Aliphatic C<sub>n</sub>H<sub>n</sub>  
 Weight % Total C, O, and H  
 Weight % CH<sub>4</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>4</sub>H<sub>8</sub>, C<sub>4</sub>H<sub>16</sub>

Char Residue, Ingredients

Weight % Total Residue  
 Weight % MgO, SiO<sub>2</sub>, ZnO, C, H, N, O

Motor Performance

Ablation Motor 1 -50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Ablation Motor 2 -50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Ablation Motor 3 -50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Regression Motor 1 -50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Regression Motor 2 -50, 100, 225, 400 Btu/ft<sup>2</sup>-sec  
 Regression Motor 3 -50, 100, 225, 400 Btu/ft<sup>2</sup>-sec

Figure 24. Variables for Correlating Ingredients, Properties, and Motor Performance

## Correlation Motors, Ablation

	Motor 1				Motor 2				Motor 3			
	50	100	225	400	50	100	225	400	50	100	225	400
Weight % C, Virgin		-0.90	(-0.84)		(-0.81)	-0.89				-0.95	(-0.93)	
Weight % O, Virgin		0.96	0.94			0.88	0.88			0.89	0.92	
Weight % Si, Virgin					0.89				(0.85)	(0.81)		
Weight % SiO <sub>2</sub> , Virgin					(0.87)				0.91	0.90		
Weight % H <sub>2</sub> O, Virgin					(-0.81)							
Weight % Filler, Virgin					0.89	(0.83)	(0.86)	0.88	(0.87)	0.94		(0.82)
C/O Ratio, Virgin		(-0.81)			(-0.84)	-0.91			(0.81)	-0.96		
O/H Ratio, Virgin	0.96	0.95	0.93									
Wt% H <sub>2</sub> O in Gases @ 550°C/350 psi					(-0.82)	-0.93	(-0.81)			(-0.86)		
Wt% Total H in Gases @ 550°C/350 psi				(-0.88)	(-0.87)							
Wt% CH <sub>4</sub> in Gases @ 550°C/350 psi							-0.90	-0.95			-0.93	-0.96
Wt% C <sub>2</sub> H <sub>6</sub> in Gases @ 550°C/350 psi		(-0.84)	(-0.81)								-0.93	(-0.82)
Wt% C <sub>2</sub> H <sub>4</sub> in Gases @ 550°C/350 psi					(-0.80)				-0.90			
Wt% C <sub>4</sub> H <sub>6</sub> in Gases @ 550°C/350 psi	-0.92	-0.95	-0.89								(-0.84)	(-0.87)
Wt% SiO <sub>2</sub> in Residue		(0.86)	(0.83)			0.98	0.93		(0.87)	0.97	0.99	
Wt% ZrO in Residue		(-0.87)	-0.90				-0.91	-0.95			-0.96	(-0.87)
Wt% C in Residue					-0.92	-0.91			-0.92	-0.98		
Wt% H in Residue	-0.92	-0.93	(-0.87)						(-0.80)	-0.88	(-0.82)	

\* Critical Value @ 95% Confidence Level = 0.88

Critical Value @ 90% Confidence Level = (0.80) (data has correlation trend only)

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Correlation Motors, Regression

Motor 3				Motor 1				Motor 2				Motor 3			
50	100	225	400	50	100	225	400	50	100	225	400	50	100	225	400
	-0.95	(-0.83)			-0.94	-0.95	-0.89			(-0.86)					
	0.89	0.92			(0.87)	0.89	0.88			0.96					
85)	(0.81)			(0.86)								(0.82)			
91	0.90				(0.85)	(0.80)									
								-0.96	(-0.84)					-0.92	(-0.81)
87)	0.94		(0.82)	(0.80)	0.93	(0.85)	(0.82)								
81)	-0.90				-0.99	-0.91	(-0.87)								
					(0.82)	0.89				(0.87)					
	(-0.86)				-0.90		(-0.87)	(-0.82)	-0.94						
		-0.93	-0.96							(-0.87)					-0.91
		-0.95	(-0.82)							-0.89					
90														(-0.81)	(-0.86) -0.91
		(-0.84)	(-0.87)			-0.90									
87)	0.97	0.89				0.99	0.98		(0.81)	0.89					
		-0.96	(-0.87)			(-0.87)				-0.96					
92	-0.98				-0.96	-0.90	-0.90		(-0.80)						
80)	-0.38	(-0.83)			(-0.85)	-0.91	(-0.80)			(-0.87)					

Figure 25. Correlation of Material Ingredients with Ablation and Regression Performance

2

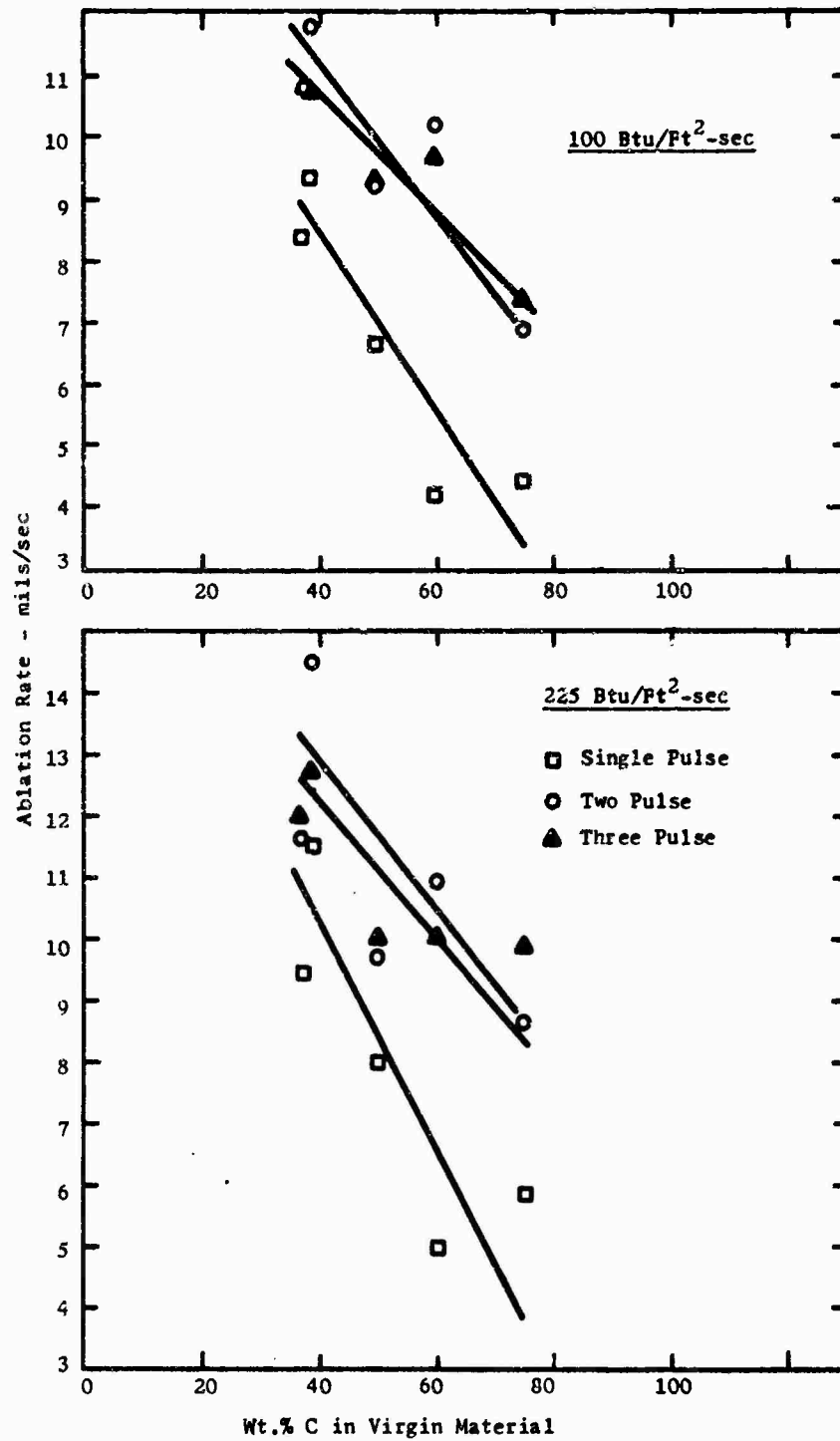


Figure 26. Ablation Rate vs Wt% C in Virgin Material

III, D, Determine Influence of Ingredients on Performance and Properties  
(Task E, Phase I) (cont.)

b. Weight % Oxygen, Virgin Material

The oxygen concentration in the five materials varied from 9 to 33% (Figure 23). The positive correlation coefficients indicate detrimental effects of oxygen on performance. This effect is further illustrated in Figure 27.

c. Ratio C/O, Virgin Material

The C/O ratio for the five materials varied from 1/1 to 8.3/1 as shown in Figure 23. The previously demonstrated relationships for carbon and oxygen concentrations with performance would seemingly indicate a relationship should exist between the C/O ratio and performance. However, the correlation coefficients obtained for these parameters only indicate a trend toward a linear relationship. The plots in Figure 28 indicate that this relationship is significant for C/O ratios from 1 to 4. A larger number of data would be required to establish a relationship at higher C/O ratios.

d. Ratio O/H, Virgin Material

The correlation coefficients indicate that a linear relationship exists between the O/H ratio and ablation rates in the single-pulse correlation motor. The plots in Figure 29 show, however, that ablation rates generally increased with increasing O/H ratio in all three motors. This indicates that a high hydrogen concentration and low oxygen concentration is desirable in the materials to obtain good performance.

e. Effects of Inorganic Filler Concentrations

Data previously obtained in insulation R&D programs at Aerojet relating wt% inorganic filler to performance generally show that ablation rates decrease with increasing concentrations of filler up to 50 wt% with the lowest rates being achieved in the range of 30 to 50 wt%. Beyond 50 wt%, the rates once again tended to increase. An examination of the data from this program (Figure 30) indicates that only at the 225 Btu/ft<sup>2</sup>-sec heat-flux level does there appear to be any general trend towards an optimum filler concentration. At this heat flux level, however, contrary to previous findings, the data still show good performance at the lower filler concentrations. It is apparent that much more data would be required to establish a relationship between wt% inorganic filler and performance.

f. Weight % Hydrogen in Char Residue

The concentration of hydrogen in the virgin materials varies from about 5 to 9 wt% in the five materials. Part of this hydrogen, from 0.5 to 1.83 wt%, was retained in the char residue. Although the concentration is low, the correlation coefficients indicate a trend toward linear

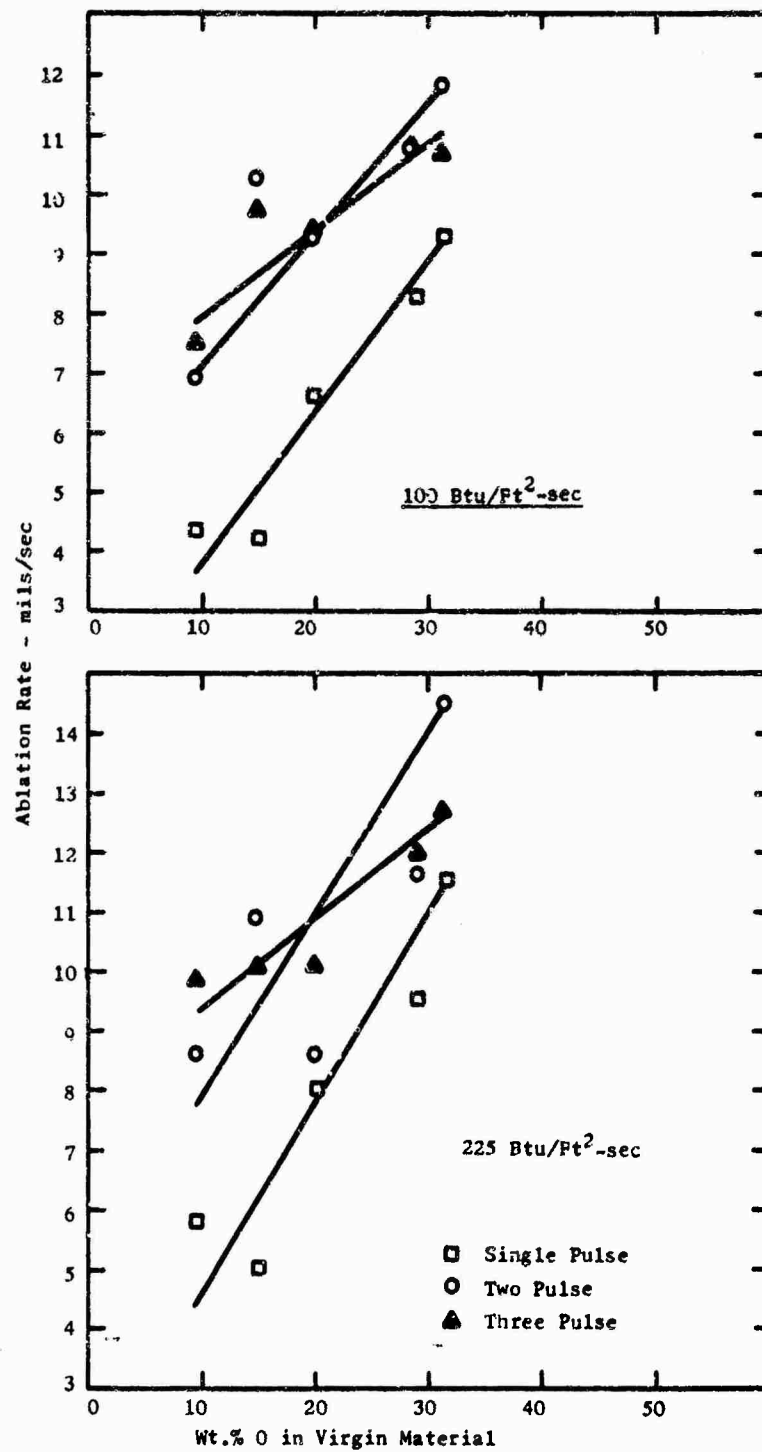


Figure 27. Ablation Rate vs Wt% O in Virgin Material

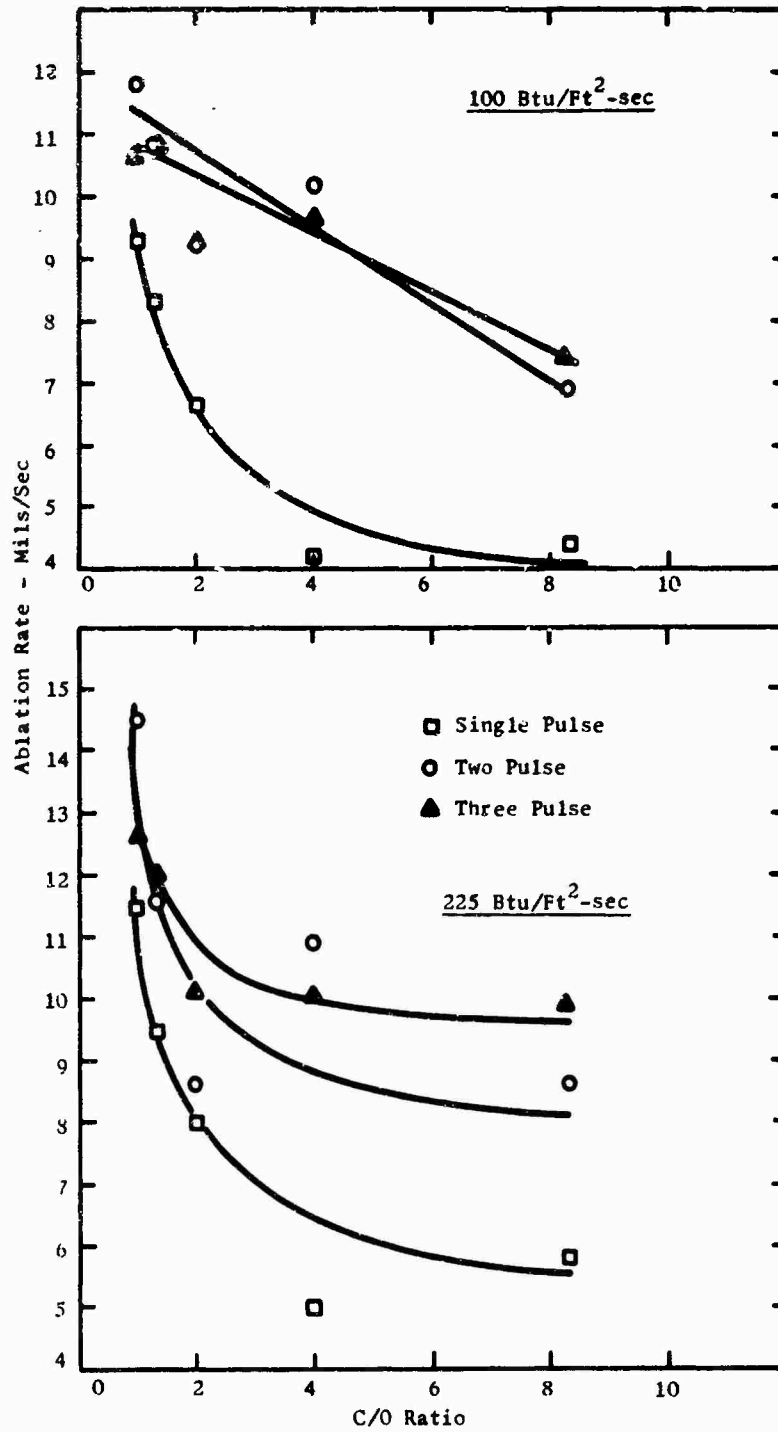


Figure 28. Ablation Rate vs C/O Ratio in Virgin Material



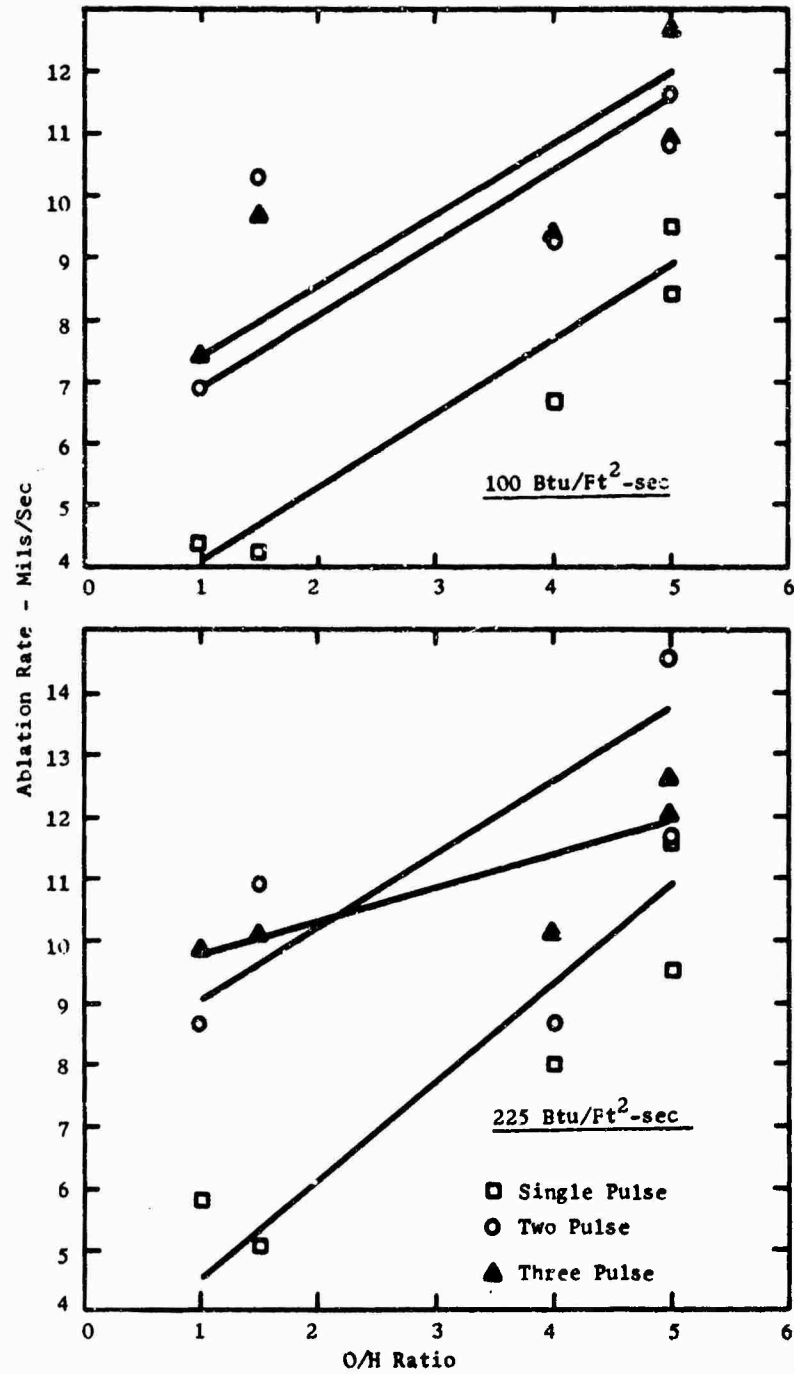


Figure 29. Ablation Rate vs O/H Ratio in Virgin Material

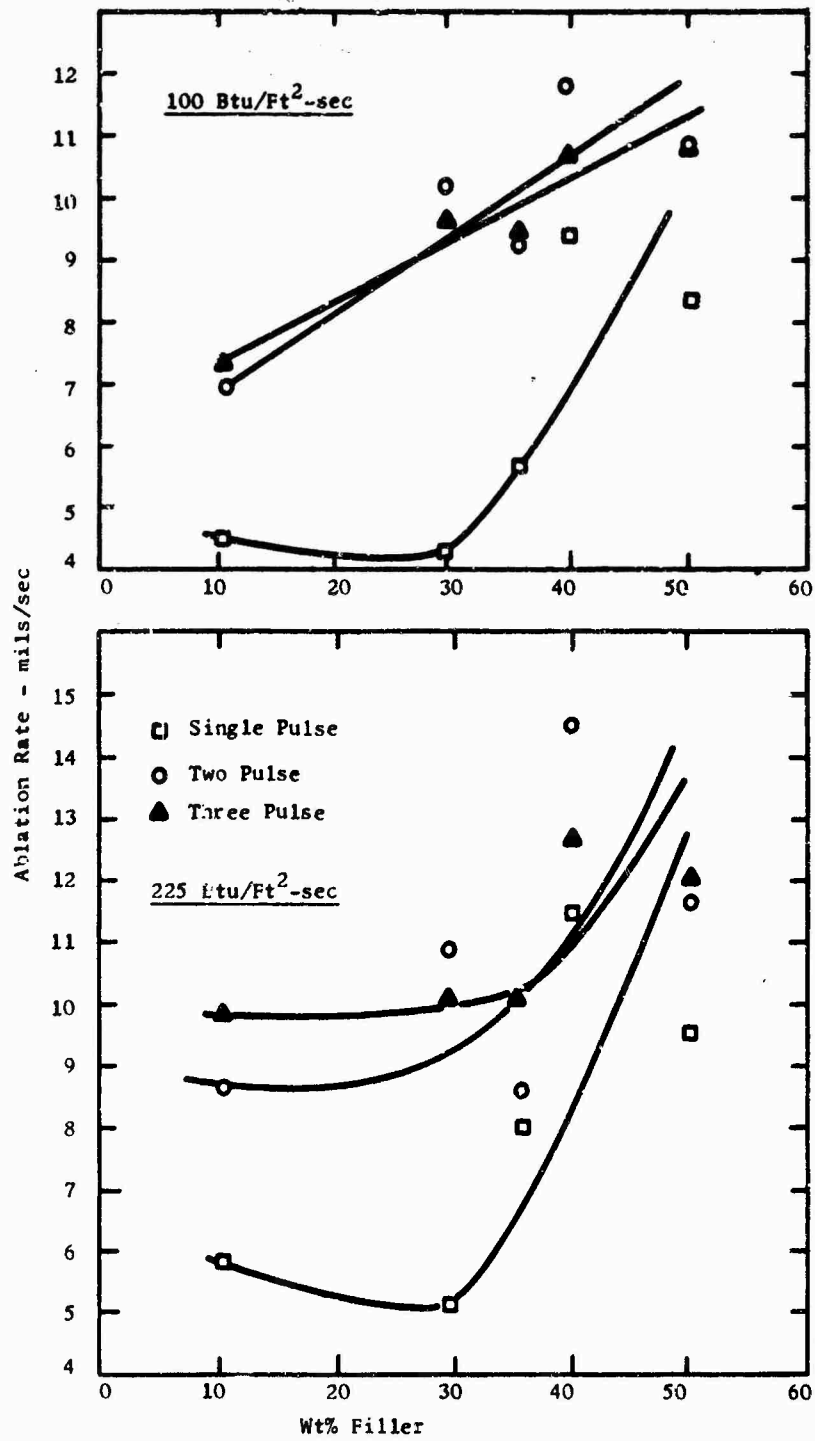


Figure 30. Ablation Rate vs Wt% Inorganic Filler

III, D, Determine Influence of Ingredients on Performance and Properties  
(Task E, Phase I) (cont.)

relationship between ablation rate and wt% hydrogen in the residue. The plots of ablation rate versus wt% hydrogen in the residue, in Figure 31, show that ablation rates decrease with increasing wt% hydrogen in all of the three motors at 100 and 225 Btu/ft<sup>2</sup>-sec.

g. Weight % Carbon, Char Residue

The wt% carbon retained in the char residue of the five materials varied from 30 wt% for V62 to 1.5 wt% for 81-8, as shown in Figure 23.

The V62 material having the highest wt% carbon in the virgin material also retains the highest wt% carbon in the char, as shown below:

<u>Wt% Carbon</u>	<u>V62</u>	<u>126K</u>	<u>V44</u>	<u>81-8</u>	<u>40SA40</u>
Virgin Material	75	60	50	37	39
Char Residue*	30	15	12.2	1.5	6.5
% Reduction	60	75	75.6	95.8	83.3
% Retained in Char	40	25	24.4	4.2	16.7

\*As related to original weight of virgin materials.

These data further show that the lower the material carbon concentration, the higher the percentage loss. The significance of retaining a high wt% carbon in the char is indicated in Figure 32, which shows a general correlation of ablation and wt% carbon concentration. The best correlation was obtained at the 100 Btu/Ft<sup>2</sup>-sec heat-flux level.

h. Weight SiO<sub>2</sub> MgO and ZrO Char Residue

The major filler ingredients of the five materials are asbestos in short fiber forms and SiC<sub>2</sub> in fiber or powder forms. The 40SA40 material contains SiO<sub>2</sub> in fiber filler and no asbestos while the other materials contain combinations of asbestos and SiO<sub>2</sub>. On decomposition, asbestos forms SiO<sub>2</sub> and MgO. Figure 23 shows that SiO<sub>2</sub> was the major ingredients in the char residue of all materials. MgO was found in the residue of all materials except 40SA40. Small concentrations of ZnO also were found in the residue of all materials except 40SA40.

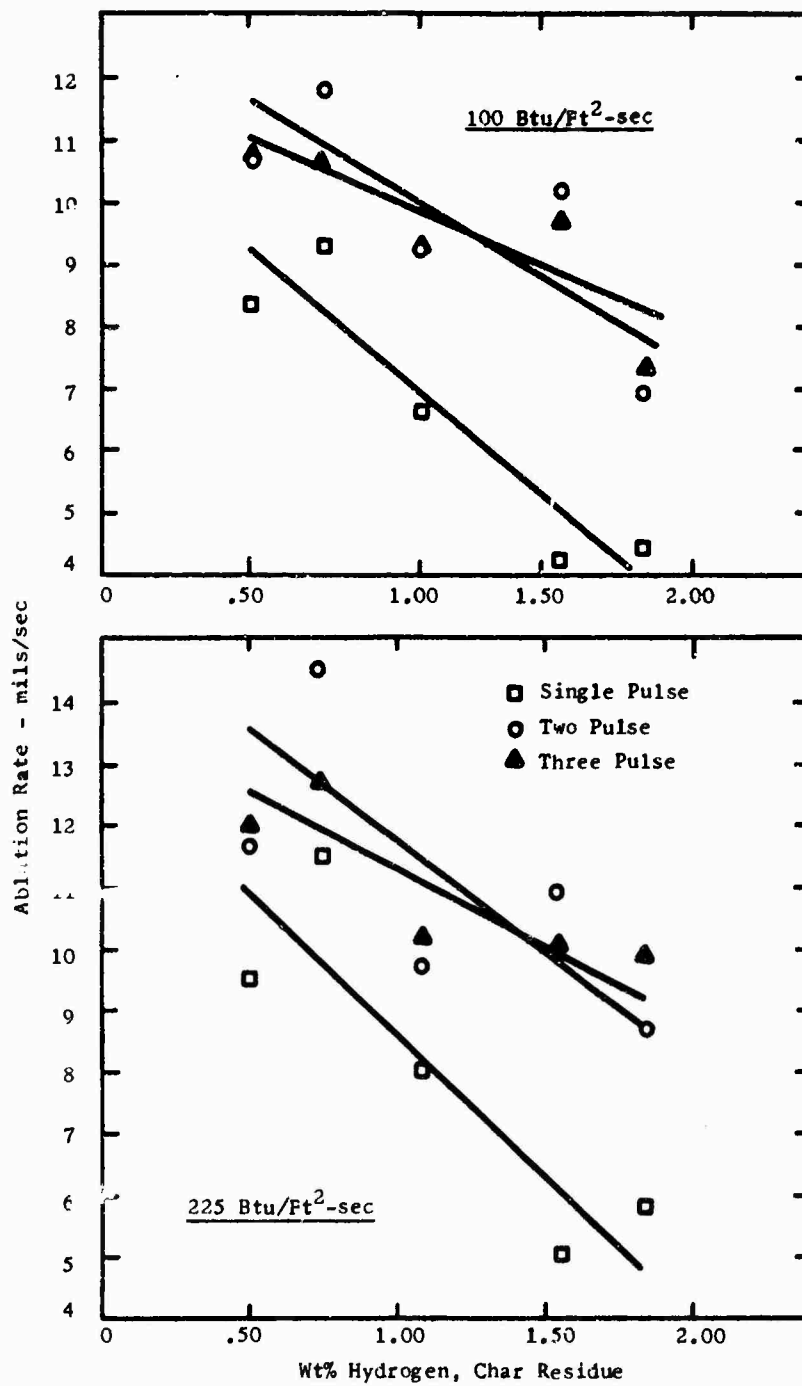


Figure 31. Ablation Rate vs Wt% Hydrogen in Residue

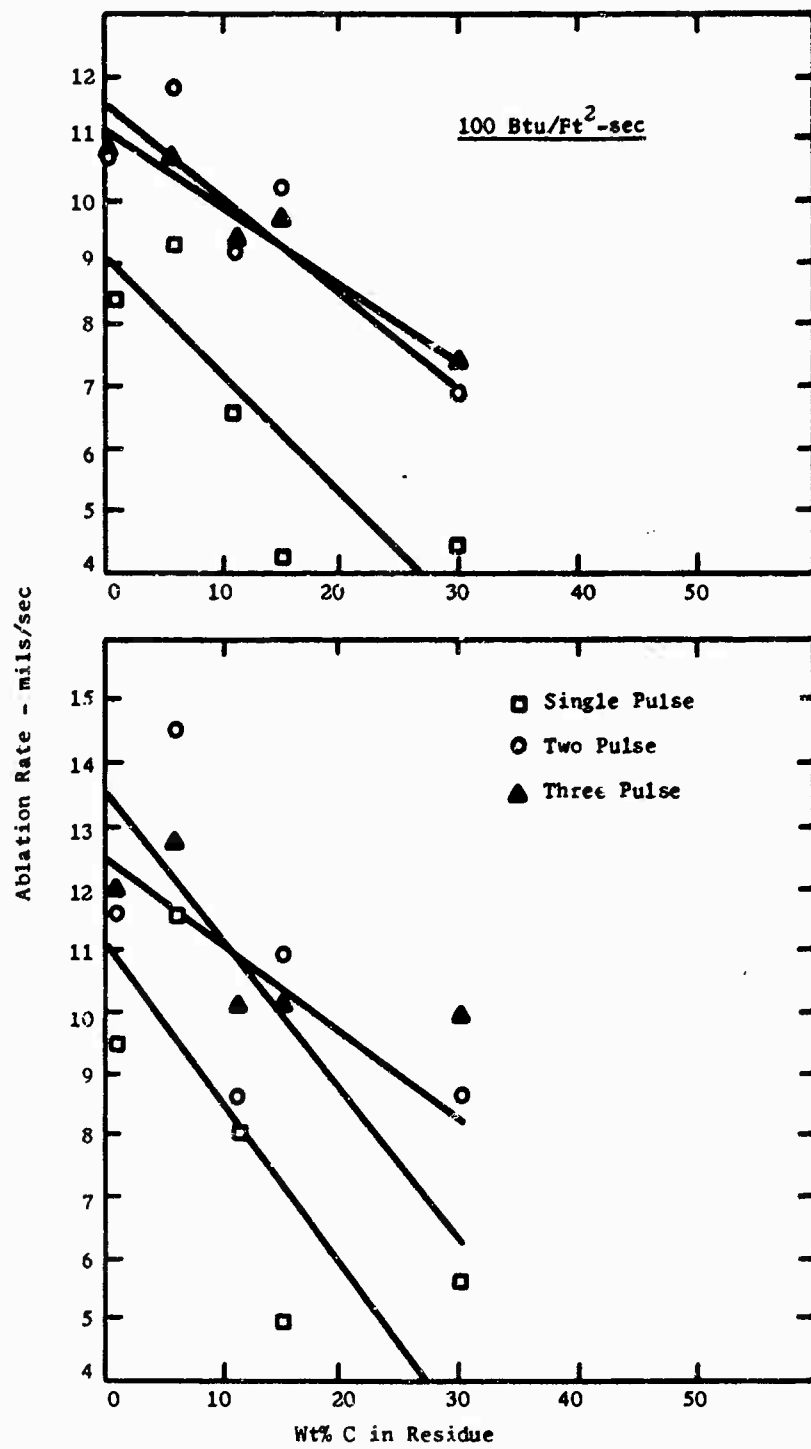


Figure 32. Ablation Rate vs Wt% C in Residue

III, D, Determine Influence of Ingredients on Performance and Properties  
(Task E, Phase I) (cont.)

The correlation coefficients indicate a relationship between performance and wt% residue ingredient for  $\text{SiO}_2$  and  $\text{ZnO}$ . However, plots of ablation rate versus wt% of ingredients in residue show that only wt%  $\text{SiO}_2$  has a significant effect on ablation performance. This significance is demonstrated in Figure 33 which shows a sharp increase in ablation rates with increasing wt%  $\text{SiO}_2$  in residue.

i. Effects on Performance of Gaseous Decomposition Products

The major portion of the gaseous products of the five materials were low molecular weight hydrocarbon, particularly methane, ethane, ethylene and butene. In addition to the hydrocarbons, some  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{CO}$ , and  $\text{H}_2$  were present, (Figure 23). The correlation coefficients indicate a trend toward linear relationship between performance and concentration of several hydrocarbons and water. However, plots of ablation rate versus wt% gaseous decomposition products indicated that these ingredients had no significant effect on performance even though it is believed they should from transpirational cooling considerations. Additional data would be required to establish effects and relationship between gaseous decomposition products and performance.

j. General Comments on Influence of Ingredients on Performance

The superior performance of the V62, SBR-phenolic material in the three motor firings can be attributed partly to its relatively high carbon concentrations, its low oxygen concentration, and to physical properties such as a high heat of combustion and low thermal conductivity. The data also indicates that its char residue, consisting of a relatively high concentration of carbon and  $\text{MgO}$  and a relatively low concentration of  $\text{SiO}_2$ , provided better protection than the residues of the other materials.

The good performance of the EPR 126K material also can be attributed to a relatively large carbon concentration and large C/O ratio. As this material formed relatively thin char layers, a question is raised as to whether a thick char layer is necessary in stop-start motors, particularly if the char is lost between pulses.

The V44 material ranked third in performance in the three motor firings. Its performance relative to V62 and 126K can be attributed to a lower carbon concentration and a higher oxygen concentration and to a lower specific heat and heat of combustion. Its relative performance also may be attributed partly to its char properties and differences in decomposition gases. The significance of these latter effects may be shown by the results of the analytical model studies being conducted as part of this program.

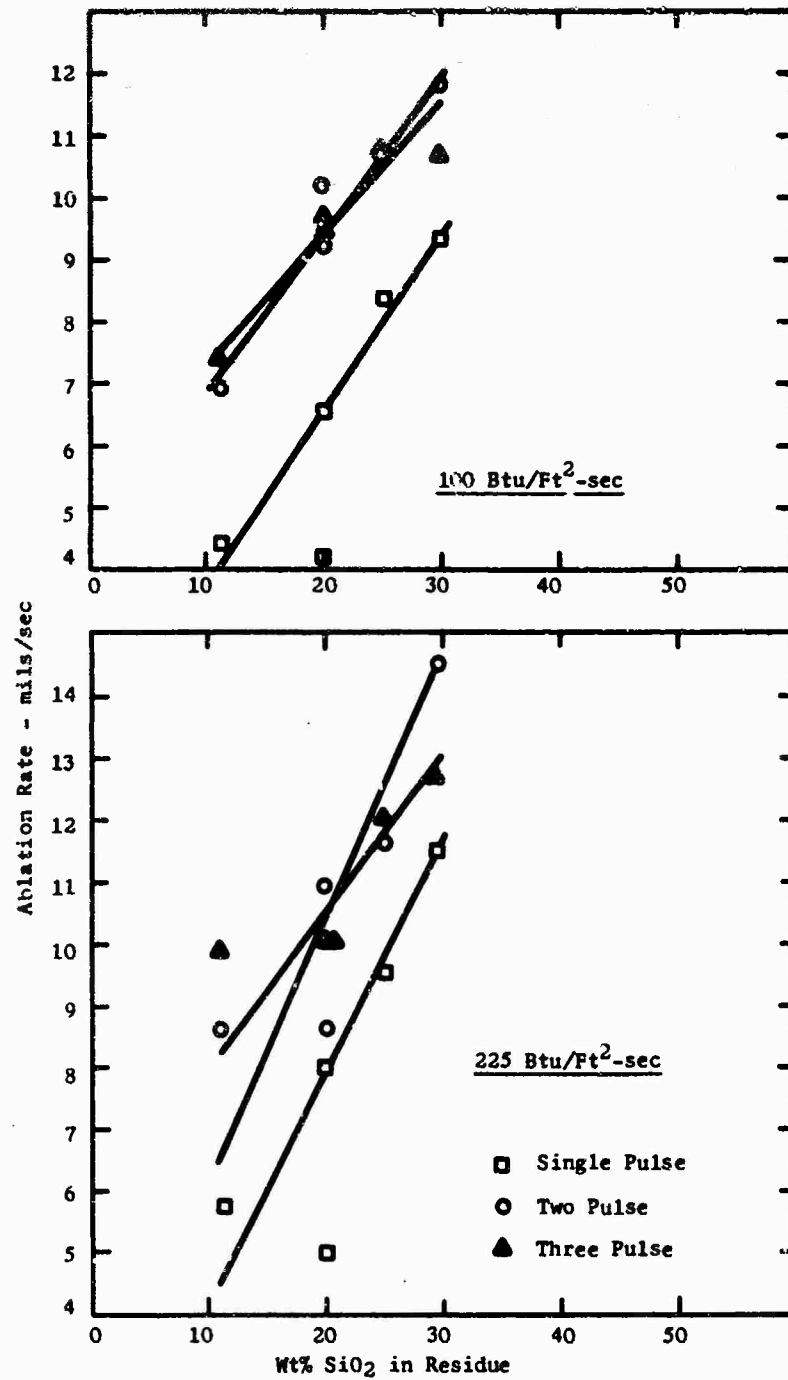


Figure 33. Ablation Rate vs Wt% SiO<sub>2</sub> in Residue

III, D, Determine Influence of Ingredients on Performance and Properties  
(Task E, Phase I) (cont.)

In comparing the performance of SMR 81-8 and 40SA40 relative to the other materials, it also seems apparent that concentrations of carbon and oxygen and their heats of combustion and heat capacities are main contributing factors. A characteristic feature of SMR 81-8 is that a large portion of its binder decomposes into gaseous molecules and its char layer consists mainly of  $\text{SiO}_2$ . The structure and properties of this char may result in ineffective transpiration cooling even though a large amount of hydrocarbon gases are present. Transpiration cooling effects in the char layer of 40SA40 also may be an insignificant factor. This material forms a thick char but a small amount of gases to provide transpiration cooling in its char.

On the basis of the results of the correlation analysis, the most significant effects of ingredients on performance can be summarized as follows:

(1) Ablation rates decrease significantly with increasing wt% carbon in the virgin material and char.

(2) Ablation rates increase significantly with increasing wt% oxygen in the virgin material.

(3) Ablation rates decrease with increasing C/O ratio, particularly in the range from 1 to 2.

(4) Ablation rates generally increase with increasing O/H ratio.

(5) Ablation rates increase significantly with increasing wt%  $\text{SiO}_2$  in the char residue.

Effects of gaseous decomposition products on performance could not be demonstrated because of an insufficient data.

2. Significant Properties

The properties of interest are those which were shown in Task C (paragraph A, above) to have significantly affected performance of the five materials in motor firings. These properties were heat capacity, thermal diffusivity, thermal conductivity and heat of combustion of the virgin materials.

The correlation coefficients for these components of the virgin materials, gas species, and char residues which have some significant relationships with one or more of the properties are shown in Figure 34.



	<u>Heat Capacity</u>	<u>Thermal Diffusivity</u>	<u>Heat of Combustion</u>	<u>Thermal Conductivity</u>
Weight % Carbon	0.990		0.983	
Weight % Oxygen	0.986	(0.849)	0.968	(0.813)
Weight % Filler	0.923		0.927	
Weight % SiO <sub>2</sub>	(-0.876)		(-0.863)	
C/O Ratio	0.942		0.944	
O/H Ratio	0.897		0.964	

\* Critical Value @ 95% Confidence Level = 0.88

Critical Value @ 90% Confidence Level = (0.80) (Data has correlation trend only)

Figure 34. Correlation between Ingredients and Properties  
of Virgin Material

III, D, Determine Influence of Ingredients on Performance and Properties  
(Task E, Phase I) (cont.)

These data indicate that heat capacity and heat of combustion are related linearly to wt% oxygen, wt% total filler, C/O ratio, and O/H ratio. A trend towards a linear relationship is also indicated for wt% SiO<sub>2</sub>. A linear relationship did not exist between ingredients and thermal diffusivity and thermal conductivity.

The manner in which heat capacity is affected by the ingredient parameters is demonstrated in Figures 35 to 37. It is noted that heat capacity increases with increasing wt% carbon and C/O ratio and decreases with increasing wt% oxygen, O/H ratio, wt% SiO<sub>2</sub>, and wt% filler. The heat of combustion is affected by the ingredients in a similar manner as the heat capacities (see Figures 38 to 40).

The correlation coefficients obtained indicate further that there was no linear relationship between ingredients and properties of ingredients such as heat of fusion and heat of vaporization.

On the basis of the results of this correlation analysis, it is apparent that the major overall ingredient parameters to consider in selecting an insulation system which will have a high heat of combustion, high heat capacity and low density and thereby superior performance are (1) a high concentration of carbon, (2) a high C/O ratio, (3) a low O/H ratio, and (4) a low concentration of oxygen in the insulation compound.

Since this analysis was limited in scope to overall ingredient parameters and only five materials, the data obtained are insufficient to distinguish between effects of molecular structure and physical forms (fiber, powder, or continuous phase binder) in which the element of carbon, hydrogen, oxygen, etc., are present in the compounds. For example, the significance of having a high concentration of carbon present only in the continuous phase binder vs being distributed as part of the binder, partly as a polymeric filler in powder form, partly as carbon black powder or, partly as carbon fiber, is not apparent from this analysis. To determine the significance of ingredient, composition, molecular structure, and form, additional data on chemical thermodynamic properties of the fillers and of bond energies, crosslink density, and polymer degradation rates would be required. Additional data also would be required to determine whether or not char properties and transpiration cooling by gaseous decomposition products in the char layers have significant effects on performance. Further specific effects of filler ingredients on the burning characteristics of the insulation after motor shutdown (between cycles) such as extinguishment by cooling, smothering and chemical reactions may be significant for the overall performance of the insulation.

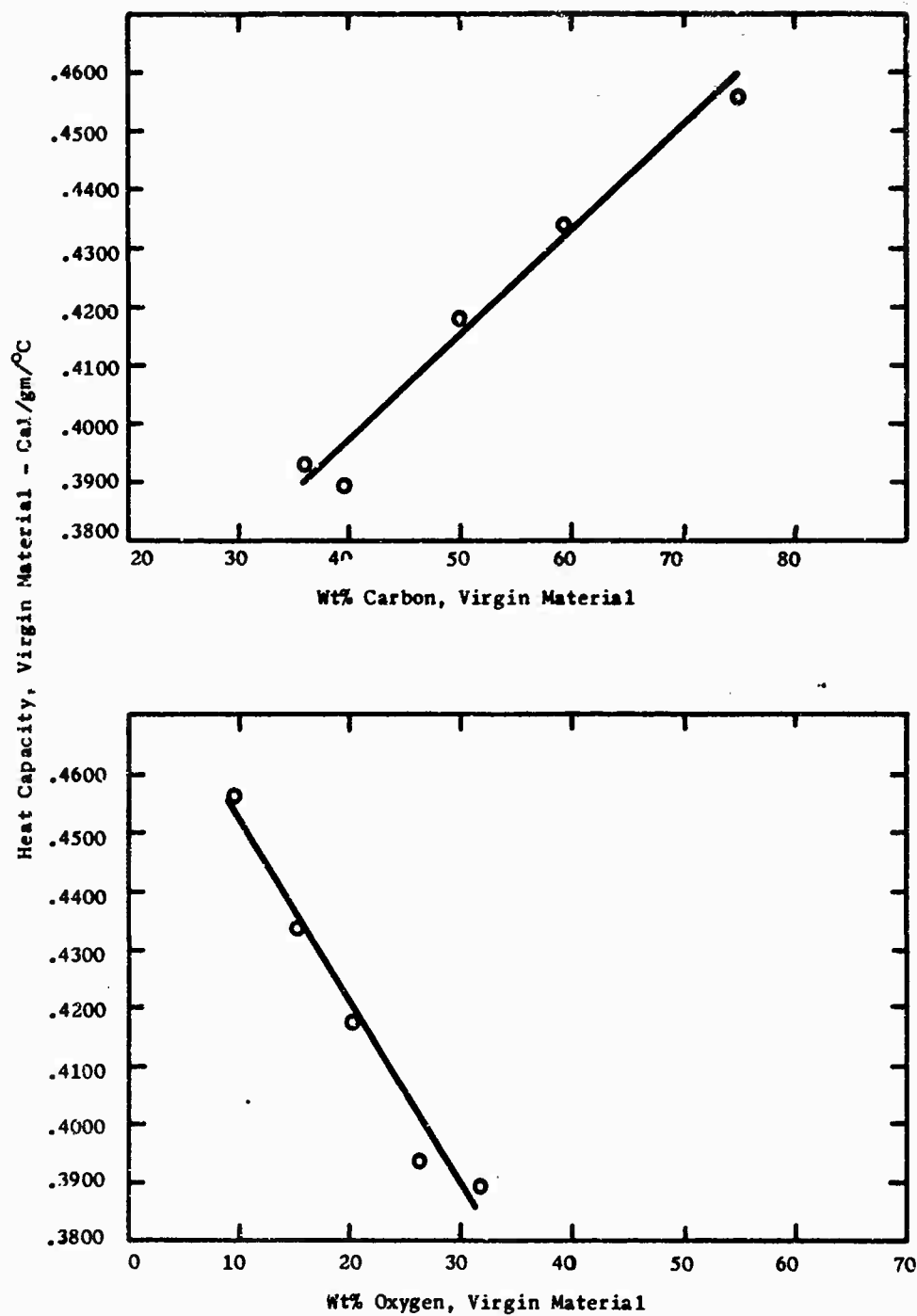


Figure 35. Heat Capacity vs Wt% Carbon or Oxygen in Virgin Material

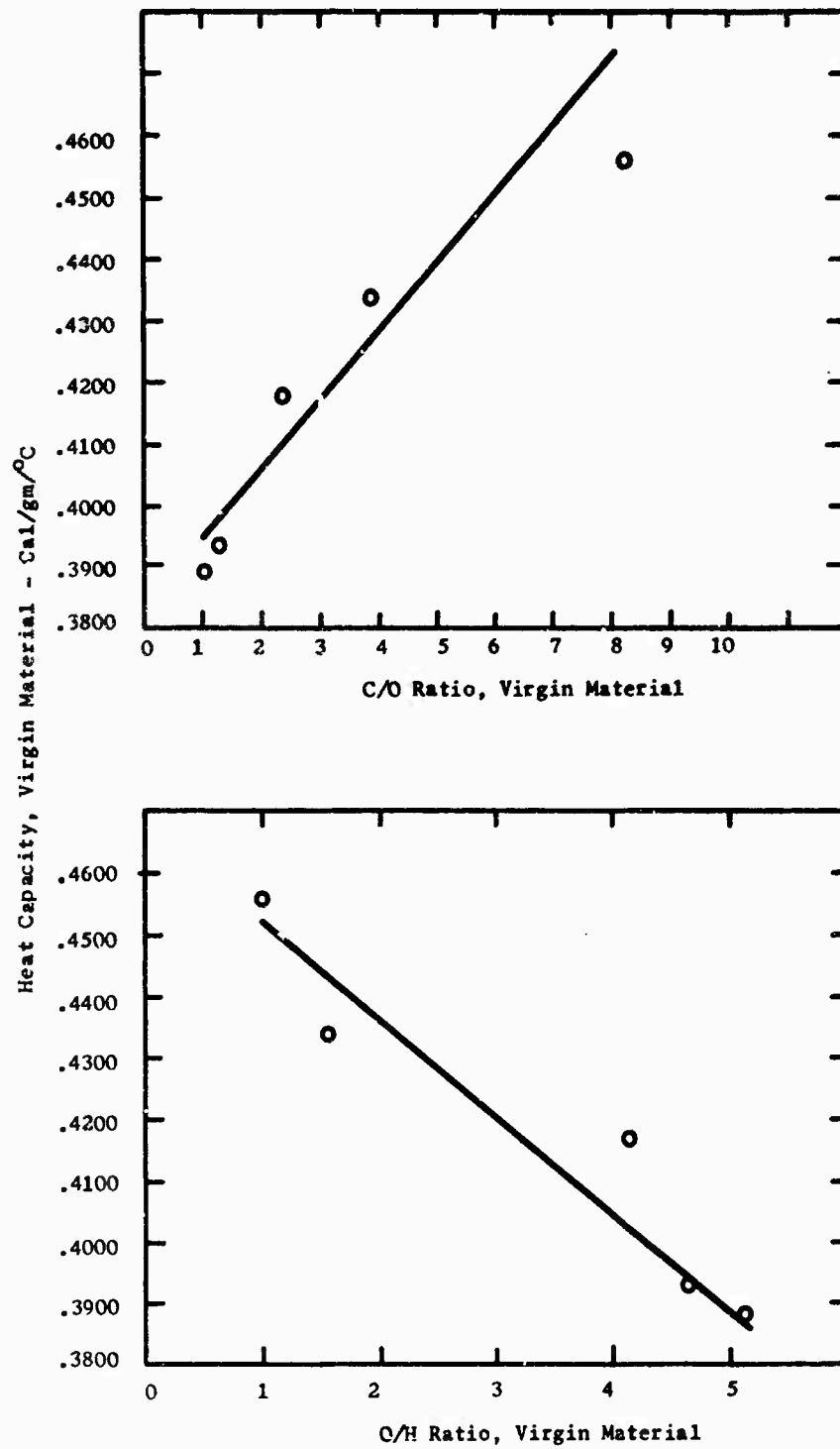


Figure 36. Heat Capacity vs C/O or O/H Ratio, Virgin Material

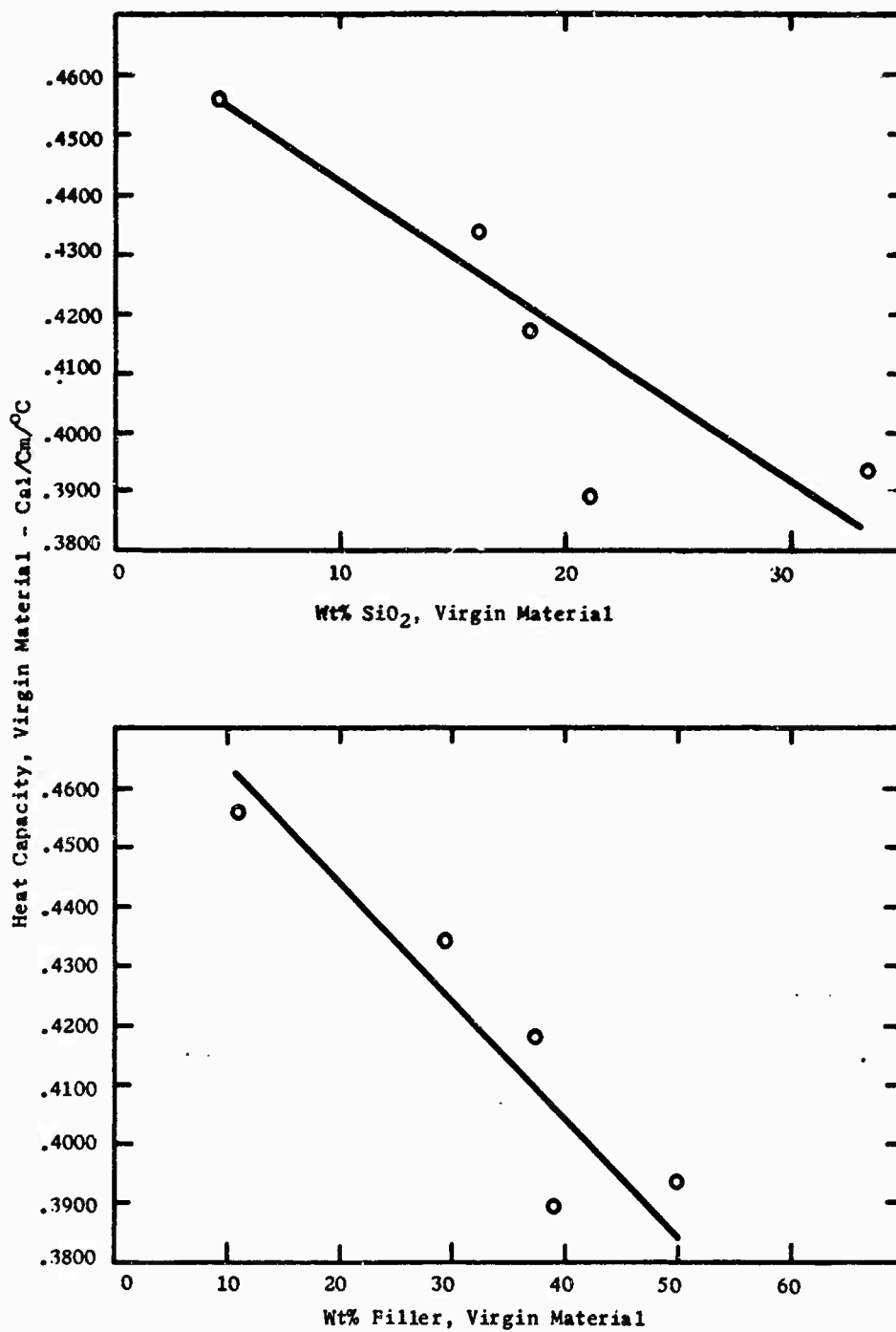


Figure 37. Heat Capacity vs Wt% SiO<sub>2</sub> or Filler, Virgin Material

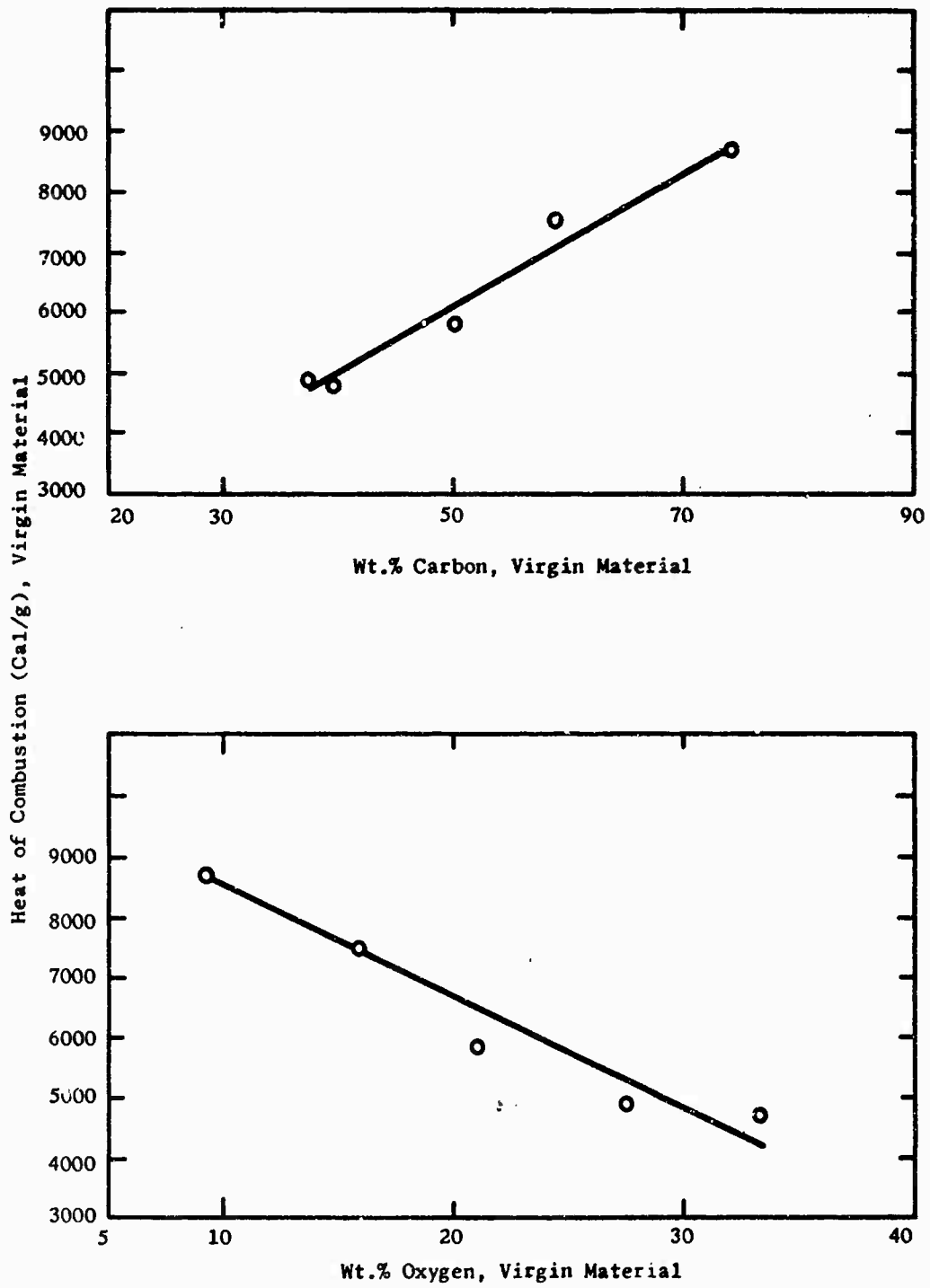


Figure 38. Heat of Combustion vs Wt% Carbon or Oxygen, Virgin Material

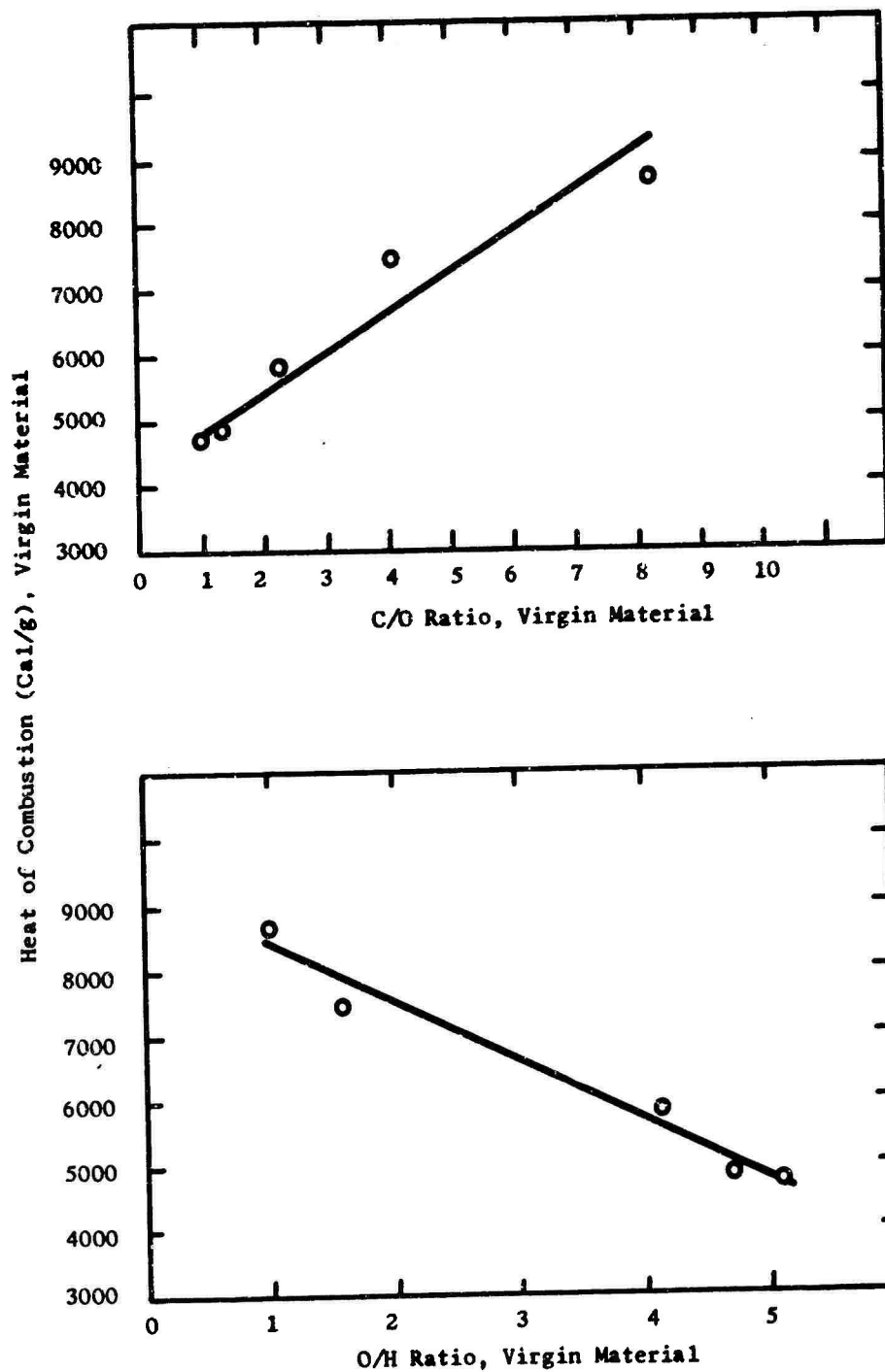


Figure 39. Heat of Combustion vs C/O or O/H Ratio, Virgin Material

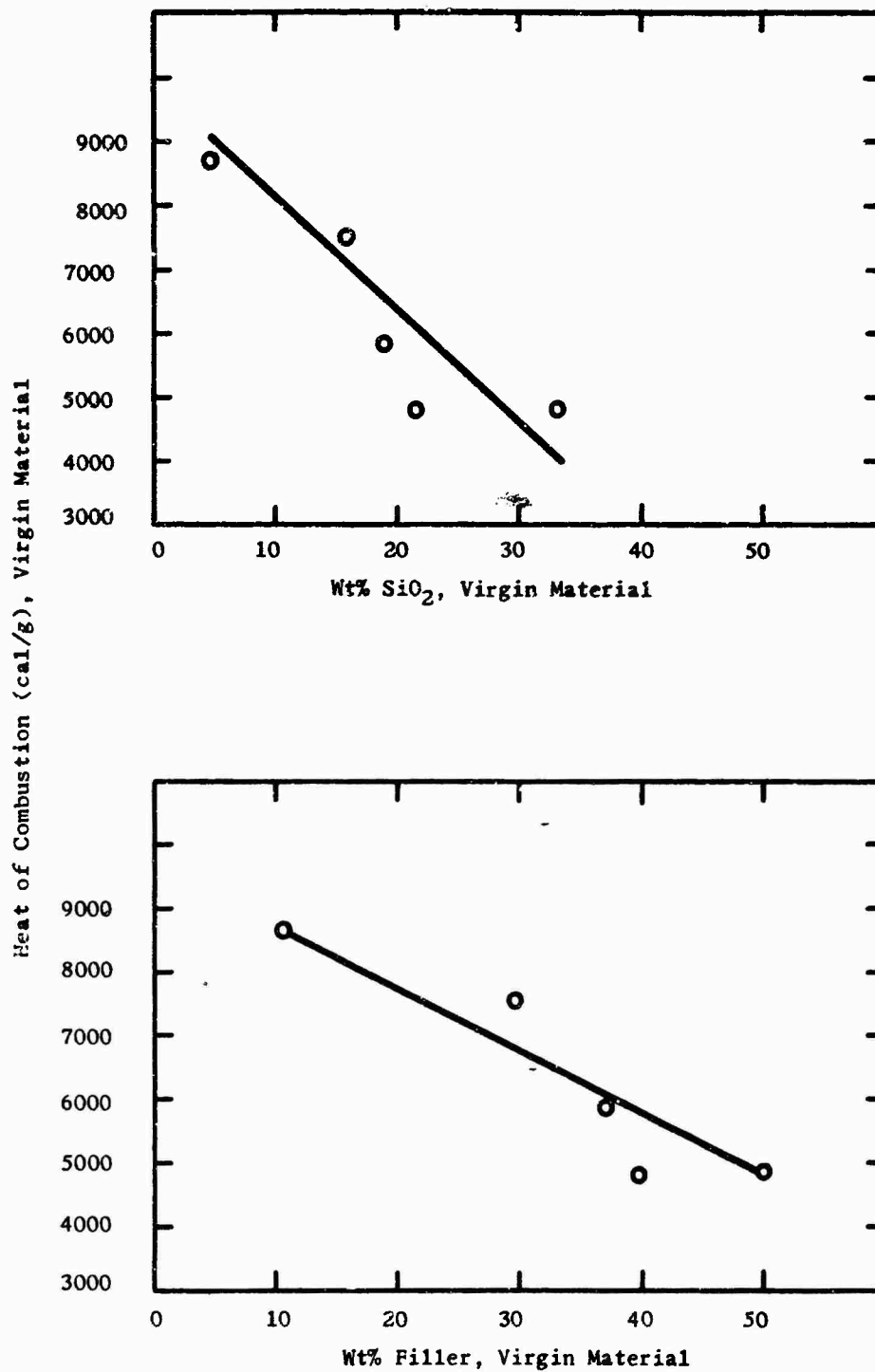


Figure 40. Heat of Combustion vs Wt% SiO<sub>2</sub> or Filler, Virgin Material



SECTION IV

WORK PLANNED FOR THE THIRD PHASE

A. ANALYTICAL STUDIES

1. Modify current analytical model for multipulse elastomeric insulation use.
2. Perform analysis of correlation motors.
3. Modify computer program as indicated by analysis in Item 2, above.
4. Predict performance of verification motors.
5. Perform analysis of verification motors.

B. VERIFICATION TESTING

1. Static test fire three verification motors (12-pulse, 60-sec total duration, 5-pulse, 100-sec total duration, and 21-pulse, 200-sec total duration).
2. Conduct postfire analysis of each motor, and compare performance of materials with that predicted during analytical studies.

APPENDIX I

CORRELATION ANALYSIS  
MULTIPLE RESTART INSULATION

I. INTRODUCTION

Data was obtained on approximately 80 variables for each of five elastomeric insulation materials. Some of these variables were measures of performance during motor firings and will be referred to as the dependent variables. The purpose of the study was to determine which of the remaining variables are related to performance and to develop equations incorporating dependent and independent variables which could be used as a screening mechanism for other candidate materials.

II. APPROACH

The variables were first screened by calculating the simple correlation coefficients using computer program AS 223U entitled "Simple Correlation Analysis." The large number of statistically significant correlations were further screened in a subjective manner for having a reasonable interpretation. Similarly, other variables which exhibited a non-significant but reasonable correlation were considered. Six independent variables were selected: density, heat capacity, thermal diffusivity, thermal conductivity, heat of combustion, and plasma-arc ablation data.

Following the selection of independent variables, single and two variable screening equations were determined by least squares using computer program AS 222U, "Multiple Regression and Correlation Analysis." The resulting single independent variable equation incorporating plasma-arc ablative data is of the following form:

$$\dot{a} = B_0 + B_1 X_1 \quad (1)$$

For those equations involving two independent variables (thermal conductivity and heat of combustion), various forms were tried such as:

$$\dot{a} = B_0 + B_1 X_1 + B_2 X_2, \quad (2)$$

$$\dot{a} = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_1^2,$$

and

$$\dot{a} = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_1 X_2$$

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Although the significance of t-values was looked at, the criteria for selecting the best of these three equations was again somewhat subjective based on a study of the residuals and the consistency of a particular form for the variables.

For those equations involving four independent variables (density, heat capacity, thermal diffusivity, and heats of combustion), a small special program was written for the IBM 1130. This was necessary because program AS 222U has a builtin restriction that the number of data points must be at least one greater than the number of parameters to be estimated. Since these equations of the form

$$\dot{a} = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 \quad (3)$$

involve five unknown parameters, and since only five data points were available, a perfect solution is obtained.

With small samples there is a high probability of obtaining quite large correlations strictly from chance. For a sample of size n the significance level of a sample correlation r, may be obtained by calculating

$$t = r / \left( \frac{1-r^2}{n-2} \right)^{1/2} \quad (4)$$

and then looking up the corresponding two-tail probabilities in tables of student's t distribution with n-2 degrees of freedom. For example, an r of 0.8 from a sample of size 5 gives a t value of 2.31 corresponding to a significance level of 10.4%. Thus, for repeated samples of size five about one in ten should be expected to yield a correlation of 0.8 or larger, when in fact the variables are completely uncorrelated. Of course, equation (4) may be set equal to a t-value corresponding to any desired probability and then solved for a critical r. Then a sample r is deemed significant if it is greater than the critical value.

Although the chance of deciding two variables are correlated when they actually are not can be made arbitrarily small by sufficiently reducing the significance level, it must be remembered that the test has very weak power with a small sample size. Thus, by reducing the significance level, the chances of deciding two variables are not correlated when they actually are, is greatly increased. The only way this probability can be reduced for a given significance level is by increasing sample size.

There is another difficulty inherent in using the simple correlations to select variables. It is easily possible for two correlated variables to appear uncorrelated until corrected for another one or more variables. Thus, variables could be thrown out which when included in a multiple correlation

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would greatly improve predictions. Although this is a difficult problem to get around at anytime, the small sample size further complicates the situation.

Even following the selection of independent variables, the use of a least squares multiple regression analysis is very restricted based on five data points. Because of the few degrees of freedom, there is essentially no way to verify the quality of the fits obtained. As the number of parameters estimated in the equation gets close to the number of data points one must necessarily get a better and better fit. Just as one can always put a straight line through any two points, a five parameter equation like (3) can be put exactly through five points. Similarly, while a three or four parameter equation will not fit perfectly to five points, it is bound to fit very well. While such equations will predict well at those particular conditions, they may well be worthless for either interpolation or extrapolation. The only safeguard in this situation is to ensure that the equations seem to have a sensible interpretation and if possible to obtain additional points for comparison with predicted values.

Because of the limitations resulting from the small sample size, the correlation analysis and accompanying equations were looked upon generally as an arbitrary screening mechanism subject to an overall technical appraisal of the results.

Reference

1. Statistics Program Manual 2300-M08, Computing Sciences Division, Aerojet-General Corporation.

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<small>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</small>		
1. ORIGINATING ACTIVITY (Corporate method)		2a. REPORT SECURITY CLASSIFICATION
Aerojet-General Corporation Sacramento, California 95809		Unclassified
		2b. GROUP
3. REPORT TITLE		
INVESTIGATION AND EVALUATION OF MOTOR INSULATION FOR MULTIPLE RESTART APPLICATION		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)		
Second Phase Report 15 Jan 1967 to 1 April 1967		
5. AUTHOR(S) (Last name, first name, initial)		
Bradley, W.		
6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
April 1967	64	1
8a. CONTRACT OR GRANT NO.	8b. ORIGINATOR'S REPORT NUMBER(S)	
AF 04(611)-11609	AFRPL-TR-67-104	
2. PROJECT NO.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
N/A		
10. AVAILABILITY/LIMITATION NOTICES		
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11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY
None		Air Force Rocket Propulsion Laboratory Research and Technology Division Air Systems Command, Edwards, Calif.
13. ABSTRACT		
<p>The primary purpose of this program is to investigate the properties and behavior of elastomeric insulation materials during multiple restart conditions and the influence of these properties on materials performance.</p> <p>During the second phase of work, a correlation analysis was conducted to determine which virgin and charred material properties were significantly related to the performance of five representative materials during one, two, and three-pulse motor firings. These properties were then determined on ten additional materials. All 15 materials were reviewed primarily on the basis of these properties and four primary candidates (V-62, V-44, 9790VI-126K, and USR 3800) were selected for further evaluation in pulse motor firings (5, 12 and 21 pulses). A correlation analysis was also conducted to establish the relationship of the fillers, additives, and chemical ingredients to firing performance and significant properties.</p>		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Insulation Candidates Multiple Restarts. Properties of Insulation Selected Materials Ingredients						

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